Carbon Aerogels Preparation With Kaolin addition

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Abstract. Wet gels (WGs) have been obtained from the polymerization of phloroglucinol (P), resorcinol (R) and formaldehyde (F) using sodium carbonate (C) as catalyst with kaolin (KL) addition. Carbon aerogels (CAs) have been gotten through the carbonization of WGs. The porous texture properties of as – prepared samples are investigated using the surface-area analyzer. The crystal structure of CAs are studied by X-ray diffraction (XRD) and the degree of graphitization are obtained through a Raman spectrometer. Meanwhile scanning electron microscopy (SEM) are executed to observe the morphology of different samples. When the addition of 5 wt\%, the samples exhibit the high specific surface area (SSA), up to 855m\textsuperscript{2} g\textsuperscript{-1}, with a concentrated pore size distribution (PSD) of 30 nm.

1. Introduction
3D CAs have become a new porous carbon materials in recent years. The plentiful of porous structure and the excellent chemical properties have attracted more and more attention to applying in different fields, such as supports, ionic adsorbent, gas separation materials and energy storage [1-3]. Due to the low density, CAs have been adopted to aeronautical materials. Therefore, CAs will be attractive candidates for different application.

CAs were firstly prepared from R and F with C as the catalysis in the 1990s. Phenolic and aldehyde compounds are the traditional agents to prepare CAs [4]. In recent years, the emerging of different precursors have greatly improved the properties of as-obtained CAs. The precursors with N-doping are beneficial for the chemical properties; the precursors of R and F with metal ions catalysts can facilitate the improvement of SSA; the precursors containing heteroatom (B, F, P and S) could accelerate the preparation period and increase the porous structure [5,6].

The traditional CAs are low SSA, poor chemical properties and long preparation period. In consequence, the addition of heteroatom executed a pivotal role to improve the properties of CAs [7]. Kaolin are a ideal additive with multitudinous of heteroatom, and the sources are abundant and low-cost [8]. The multitudinous of heteroatom not only catalyzed the polymerization of P, R and F, but also improved the properties for CAs. Meanwhile the addition of Kaolin could well facilitate the preparation period. In this work, we obtained CAs with the addition of kaolin, the porous texture properties have been improved and the SSA is up to 855m\textsuperscript{2} g\textsuperscript{-1}. The crystal structure and the degree of graphitization are also studied.
2. Experimental

2.1. Materials
The reagents of P, R, F, C and Kaolin are all analytical grade. Ultima IV X-ray diffraction instrument (XRD, Rigaku, Japan), Specific surface analyzer ASAP2420 (micromeritics, USA), scanning electron microscope (SEM) and a Labram HR evolution Raman spectrometer (532 nm).

2.2. Preparation of CAs
CAs are gotten from the polycondensation of P, R and F with the addition of kaolin, using C as catalysts and deionized water as the solvent. The P was 0.05 mol, the R was 0.33 mol, the F was 0.76 mol, the C was 0.76mmol, and the KL was 0g, 1.06g (2.5 wt%), 2.13g (5 wt%), 3.2g (7.5 wt%) and 4.26g (10 wt%), respectively. The deionized water was 120 ml. P, R, F, C, and KL were added into deionized water to get a solution in test tubes. These tubes were kept 50℃ for some time until the hydrogels were obtained. The dried aerogels were carbonized at 900℃ for 3 h with a heating rate of 2℃/min under a flowing nitrogen atmosphere (100 mL/min).

3. Results and discussion

The N₂ adsorption–desorption isotherms of CAs are obtained to investigate the porous texture properties and shown in Figure 1. Obvious, the hysteresis-loop can be observed, which indicates the presence of mesoporous structure. The result exhibits all the samples with plentiful porous structure. And as shown in Figure 2, the PSD curves are concentrated and the pore size are all less than 50 nm, which are the mesopore, this result is in accordance with N₂ adsorption–desorption isotherms analysis.

As listed in table 1, the parameters of texture properties are obtained from the surface-area analyzer. Compared with different addition amount, when the addition is 5 wt%, the SSA is up to 855m² g⁻¹. When the addition is more than 5 wt%, KL would effect the formation of micropores and the SSA of CAs would lower to 625m² g⁻¹ and 553 m² g⁻¹. The pore size also is affected by the addition amount of KL, the more addition can decrease the pore size and the less addition can enlarge the pore size.

| Entry | S_BET (m²/g) | S_micro (m²/g) | D_average (nm) | V_total (cm³/g) |
|-------|--------------|---------------|----------------|-----------------|
| CA-1  | 591          | 424           | 5.44           | 1.013           |
| CA-2  | 664          | 426           | 5.60           | 0.875           |
| CA-3  | 855          | 536           | 5.98           | 1.267           |
| CA-4  | 625          | 390           | 6.24           | 0.978           |
| CA-5  | 553          | 363           | 6.83           | 0.941           |

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Figure 3 XRD patterns

Figure 4 Raman spectra of CAs

The crystal structure are studied through the XRD patterns and the results are presented in Fig. 3. As shown in Figure 3, the diffraction peak of 24° (2θ) is the (002) reflections and the diffraction peak of 42°(2θ) is the (101) reflections, which is corresponding to graphite carbon diffraction and crystal plane diffraction, respectively. As seen in Figure 4, the Raman spectra are obtained for different samples. Obviously, the two peaks can be observed, which is at at 1370 cm⁻¹ and 1595 cm⁻¹, respectively. These two peaks are assigned to graphite carbon (G line) and amorphous carbon (D line), respectively. The degree of graphitization of the different as-obtained CAs were calculated from ID/IG: 1.72, 1.77, 1.89, 1.82 and 1.79. The addition amount of KL is 5 wt%, the graphitization of CAs is the highest, which indicated the addition of KL can the graphitization.
Figure 5 SEM images of the CAs

The SEM images of CAs are obtained and presented in Figure 5. As shown in Figure 5, the interconnected porous structure with abundant irregular polymer nanoparticles can be clearly observed. Compared with others, CAs-3 exhibited a more well interconnected porous structure. The addition of KL is conducive to the formation of irregular polymer nanoparticles to construct the interconnected porous structure.

4. Conclusions

P-R-F CAs with different addition amount of KL are prepared and the porous texture properties also have been systematically investigated. When the addition amount is 5 wt%, the SSA is up to 855m² g⁻¹ and CAs-3 with a plentiful of porous structure exhibits well crystal structure and degree of graphitization. This work opens a new avenue for obtaining CAs with high SSA and excellent chemical properites by an addition method and also broadens the potential applications of KL.

Acknowledgments

This work was financially supported by the Science and Technology Program of Hebei Academy of Sciences (20705, 20706 and 201503).

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