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Pore-selective analysis of disordered and complex porous carbon materials by molecular masking and ^{129}Xe -NMR spectroscopy

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Xenon isotope–nuclear magnetic resonance (^{129}Xe -NMR) spectroscopy is a powerful technique for investigating the pore sizes of porous carbon materials, particularly those with narrow pore size distributions. However, the ^{129}Xe -NMR spectra of complex and disordered porous materials often exhibit peak broadening and overlapping owing to broad pore size distributions and the presence of differently shaped pores (*e.g.*, slit-shaped and cylindrical). This in turn makes accurate assessment of the pore sizes difficult. To overcome this issue and enable selective and individual evaluations for each pore, this study proposes a combined method based on molecular masking and ^{129}Xe -NMR spectroscopy. Fullerene C_{60} was used as the molecular masking agent. A model porous carbon material, comprised of a mixture of microporous and mesoporous carbons, was vacuum-impregnated with a C_{60} –toluene solution. Subsequently, the toluene was removed, resulting in the selective masking of micropores smaller than 1 nm by the C_{60} molecules. In addition, ^{129}Xe -NMR spectra were measured at $-75\text{ }^{\circ}\text{C}$ under a Xe gas pressure of 100 kPa for the model porous carbon material before and after the C_{60} masking. These spectra showed that C_{60} masking totally eliminated the spectral contributions from micropores smaller than 1 nm, while leaving the signals from larger mesopores essentially unchanged, enabling an individual evaluation of unmasked mesopores. N_2 adsorption and desorption isotherms confirmed these observations. Taken together, these results demonstrate the potential of the combined method for selectively evaluating the pore sizes of disordered and complex porous carbon materials.

KEYWORDS: Amorphous porous carbon, Combined application, Fullerene, Selective evaluation, ^{129}Xe -NMR

1. Introduction

Accurately evaluating the pore structure, including the size, shape, and connectivity, of amorphous porous carbon materials is essential for optimizing their use in adsorption, separation, and electrochemical energy storage [1–5]. However, methods such as transmission electron microscopy and X-ray diffraction are not suitable for obtaining clear information on the pore structures of amorphous porous materials, unlike those of crystalline porous materials [6–9].

Xe has long been regarded as a useful and non-destructive molecular probe because its large and spherically symmetric electron cloud is highly sensitive to its surrounding environment. Therefore, Xe isotope–nuclear magnetic resonance (^{129}Xe -NMR) spectroscopy can provide detailed information regarding the environment around a

given Xe molecule [10–12]. In our previous study [13], we designed and constructed a temperature- and pressure-controlled ^{129}Xe -NMR system and re-evaluated the interactions between pore wall and adsorbed Xe molecules using a series of KOH-activated carbon samples with narrow pore size distributions. This study revealed that the interactions between pore wall and Xe molecules depended only on a parameter related to the pore size of the activated carbons and were independent of the content of oxygen-containing surface functional groups. These findings confirmed the applicability of adsorption-controlled ^{129}Xe -NMR spectroscopy as a pore size evaluation method for amorphous porous carbon materials without assuming the pore shape.

However, peak broadening and overlapping in the NMR spectra limit the practical application of ^{129}Xe -NMR spectroscopy for disordered and complex porous carbon materials with broad pore size

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distributions and multiple pore types [14–17]. Therefore, to establish ^{129}Xe -NMR spectroscopy as a pore-structure evaluation method applicable to various porous materials, it is necessary to solve an important issue—how to selectively analyze individual pores in disordered and complex porous materials. To this end, we combined the molecular masking [18, 19] and ^{129}Xe -NMR methods in the present study.

We selected fullerene C_{60} as a molecular masking agent because of its π -conjugated structure and strong π - π interactions with graphitic domains [20]. Moreover, C_{60} can dissolve readily in organic solvents such as toluene [21–24], which enables the solution to be impregnated into porous materials and thus provides a simple and effective route to mask pores [25, 26].

In this study, we demonstrate the combined application of molecular masking and ^{129}Xe -NMR spectroscopy as a selective pore-structure evaluation method for disordered and complex porous carbon materials. We impregnated model porous carbons with a C_{60} -toluene solution under vacuum. Subsequently, toluene was evaporated, leaving C_{60} to totally mask micropores smaller than 1 nm. The ^{129}Xe -NMR spectra of the C_{60} -masked porous carbons indicated that C_{60} masking could completely remove the spectral overlap from micropores smaller than 1 nm, enabling independent analysis of the structural features of pores larger than 1 nm. The obtained results highlight the potential of the combined method for evaluating pore structures in disordered and complex porous carbon materials.

2. Experimental

2.1 Preparation of model porous carbon materials and C_{60} masking experiments

We used porous carbons with different pore size distributions as model materials. A spherical phenolic resin (BEAPS; Particle diameter: 8 μm ; ASAHI YUKIZAI Co., Ltd., Japan) was heat-treated at 600 $^{\circ}\text{C}$ in N_2 gas at a flow rate of 200 mL/min for 1 h to yield a carbonized product (abbreviated as B6). Thereafter, B6 was used as a model microporous carbon material with a pore size less than 1 nm. MJ(4)030 (CNovel[®]; Toyo Tanso Co., Ltd., Japan), which has a pore size range of 6–60 nm, was used as a model mesoporous carbon material. In addition, B6 and MJ(4)030 were mixed in a mortar at a mass ratio of 2 : 1 to obtain a mixture named B6–MJ(4)030, which was used as a model for disordered and complex porous carbon materials containing both micropores and mesopores.

All three model porous carbon materials were subjected to C_{60} masking by the vacuum impregnation method. Fig. 1 shows the workflow of the C_{60} masking experiment for porous carbon materials. The porous carbons were first vacuum dried at 150 $^{\circ}\text{C}$ for 2 h to remove adsorbed moisture. Then, they were placed under a glass cover and maintained under vacuum while a C_{60} -toluene saturated solution (the saturation concentration of C_{60} in toluene is approximately 3 mg/mL) was continuously dropped onto the materials at room tempera-

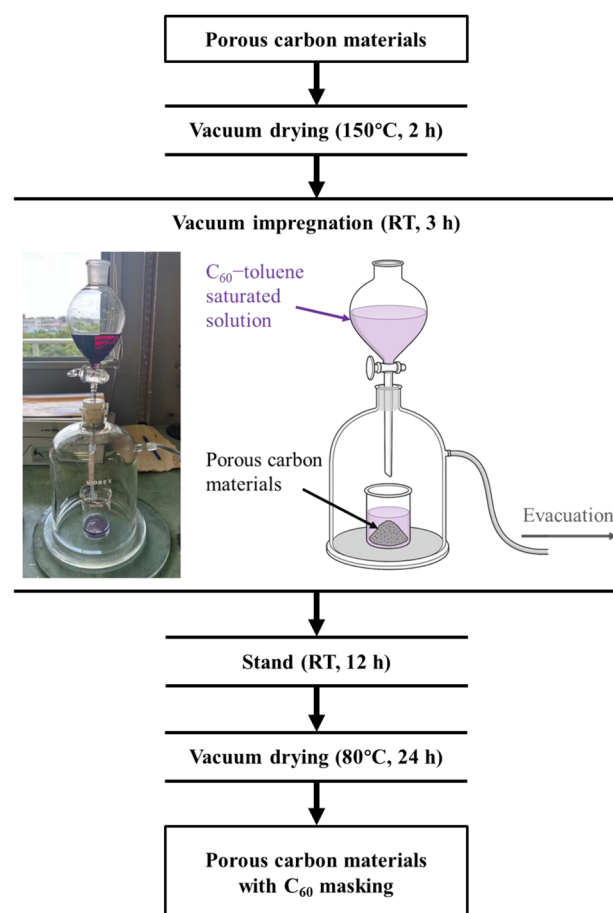


Fig. 1 Schematic of the experimental workflow for C_{60} masking of the porous carbon materials.

ture (RT) for 3 h. The impregnation solution comprised a saturated solution of C_{60} (99.95%, FUJIFILM Wako Pure Chemical Co., Ltd., Japan) in toluene. Next, the materials were allowed to stand for 12 h at RT, and the mixture, including the remaining C_{60} -toluene solution, was finally vacuum-dried at 80 $^{\circ}\text{C}$ for 24 h without any filtration step, yielding the C_{60} -masked porous carbon materials. After C_{60} masking, B6, MJ(4)030, and B6–MJ(4)030 were named B6/ C_{60} , MJ(4)030/ C_{60} , and B6–MJ(4)030/ C_{60} , respectively. For each porous carbon material, the saturated filling amount of C_{60} was calculated using the following equation:

$$\begin{aligned} &\text{C}_{60} \text{ saturated filling amount [g/g]} \\ &= \text{Pore volume of porous carbon [cm}^3\text{/g]} \\ &\quad \times \text{Density of C}_{60} (1.65 \text{ g/cm}^3) \end{aligned} \quad \dots\dots\dots (1)$$

The mass ratios of porous carbon to C_{60} used in this study are summarized in Table S1.

2.2 Gas adsorption measurements

N_2 adsorption and desorption isotherms were measured at $-196\text{ }^{\circ}\text{C}$ (77 K) using a volumetric gas adsorption measurement apparatus (Belsorp Max; MicrotracBEL Corp., Japan). The specific surface area was calculated using the α_s -plot method based on the N_2 adsorption isotherms [5, 27]. Total pore volume including that of the mesopores

was calculated from the adsorbed amount of N₂ at the relative pressure (P/P_0) of 0.99 using the following equation [28]:

$$\text{Total pore volume [cm}^3\text{/g]} = \frac{m_{\text{N}_2}}{\rho_{\text{liq.N}_2}} = \frac{M_{\text{N}_2} \cdot V_a / V_m}{\rho_{\text{liq.N}_2}} \quad \dots\dots\dots (2)$$

where m_{N_2} [g/g] is the adsorbed amount (weight) of N₂ at $P/P_0=0.99$, $\rho_{\text{liq.N}_2}$ is the density of liquid N₂ (0.808 g/cm³), M_{N_2} is the molecular mass of N₂ (28.02 g/mol), V_a [cm³(STP)/g] represents the adsorbed amount (volume) of N₂ at $P/P_0=0.99$, and V_m is the molar volume of gas at standard temperature and pressure (in this study, 22,414 cm³ (STP)/mol). The pore size distribution was obtained using non-local solid density functional theory, assuming the presence of cylindrical pores and employing the no-assumption distribution function with a solid-defined pore size, as implemented in the analysis software of the gas adsorption measurement apparatus used in this study [29].

2.3 ¹²⁹Xe–NMR spectroscopy

Using our self-designed temperature- and pressure-controlled ¹²⁹Xe–NMR system [13], the adsorption-controlled ¹²⁹Xe–NMR spectra were measured at -75°C and 100 kPa. Spectra were obtained using an NMR spectrometer (JNM-ECX400 (9.4 T); JEOL Ltd., Japan) at a frequency of 110.57 MHz for ¹²⁹Xe, with a T10 liquid probe. The sample temperature was controlled using a cryostat. ¹²⁹Xe single-pulse spectra were acquired using a 45° pulse (9.75 μs), with a recycle interval of 1.2 s. For B6 and MJ(4)030, 1024 scans were accumulated. Meanwhile, we increased the number of scans from 1024 to 4096 for B6–MJ(4)030 and B6–MJ(4)030/C₆₀ to obtain clearer spectra with reasonable signal-to-noise ratios. Chemical shifts were measured with reference to free gaseous Xe (*i.e.*, $\delta_0=0$).

3. Results and Discussion

Fig. 2 shows the N₂ adsorption and desorption isotherms and pore size distributions of B6 and MJ(4)030 before and after C₆₀ masking.

To enable a fair comparison of the changes in pore structure before and after masking, it is necessary to remove the mass contribution of the masking agent (C₆₀). Therefore, the ordinate axes (N₂ adsorption amount and pore volume change rate, dV/dd) were calculated on a weight basis for each porous carbon material. As C₆₀ itself hardly adsorbs N₂ (Fig. S1), we can exclude the possibility of a significant contribution from C₆₀ in Fig. 2. For the carbonized product B6, which possesses narrow micropores only, the N₂ adsorption amount and pore volume became nearly 0 after C₆₀ masking; this indicates that all pores of B6 were effectively masked (B6/C₆₀). Conversely, for the mesoporous carbon material MJ(4)030, with a pore size range of 6–60 nm, the N₂ adsorption and desorption isotherms and pore size distribution curve showed almost no change after C₆₀ masking. This observation indicates that mesopores larger than 6 nm were not masked (MJ(4)030 vs. MJ(4)030/C₆₀). These results indicate that C₆₀ can completely mask micropores smaller than 1 nm but has almost no effect on mesopores larger than 6 nm. This can be interpreted based on the balance of intermolecular interactions [22]. The interactions between C₆₀ and micropores smaller than 1 nm were stronger than those between C₆₀ and toluene; as a result, the micropores were completely masked by C₆₀. However, for mesopores larger than 6 nm, the interactions were much weaker, and the C₆₀ molecules remained dissolved in toluene, leaving the mesopores empty (unmasked).

Next, to investigate whether C₆₀ can function as a pore-size-selective masking agent, we performed the C₆₀ masking process using a mixture of microporous B6 and mesoporous MJ(4)030 (*i.e.*, B6–MJ(4)030) as a model bimodal porous carbon material. Fig. 3 shows the N₂ adsorption and desorption isotherms and pore size distributions of the mixture B6–MJ(4)030 before and after C₆₀ masking. Again, the ordinate axes were normalized to the mass of B6–MJ(4)030 alone. As shown in Fig. 3(a), due to the presence of micropores in B6, B6–MJ(4)030 exhibited a steep N₂ uptake at very low P/P_0 , displaying a

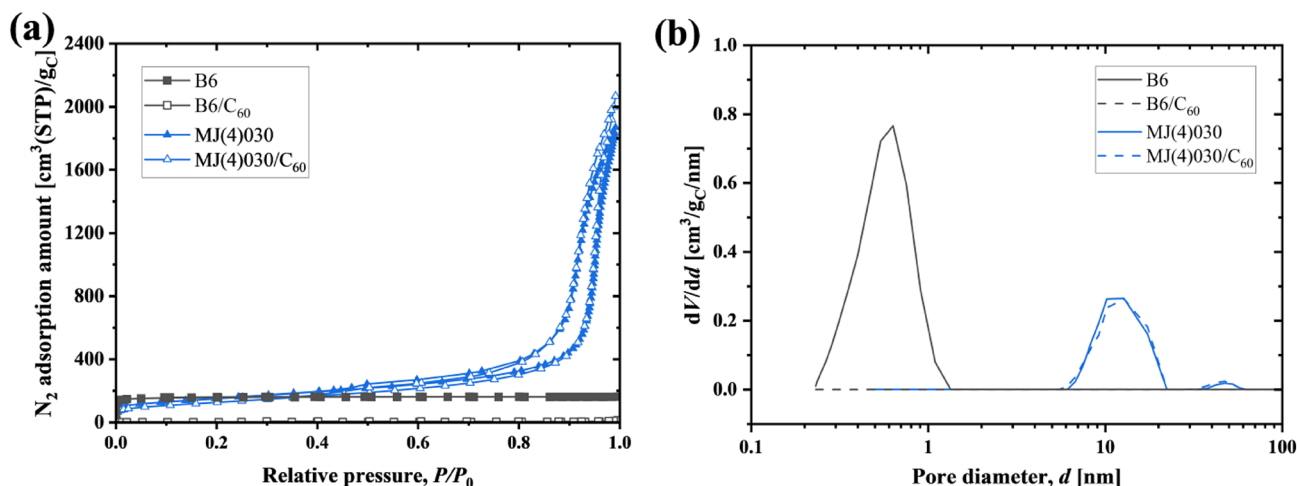


Fig. 2 (a) N₂ adsorption and desorption isotherms at -196°C and (b) pore size distribution of B6 and MJ(4)030 before and after C₆₀ masking. The ordinate axes were calculated on a weight basis for each porous carbon material.

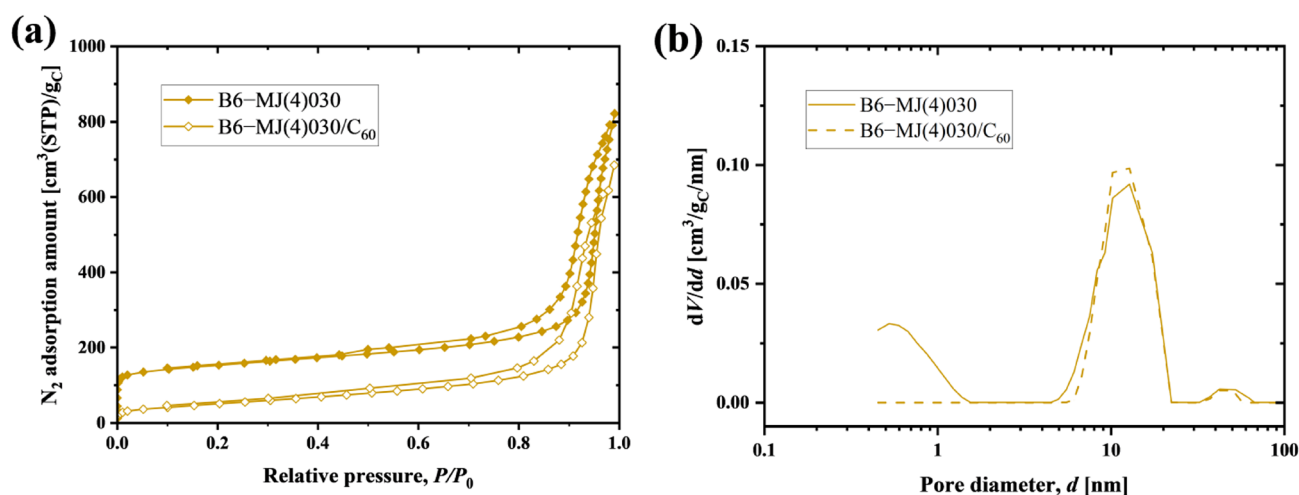


Fig. 3 (a) N₂ adsorption and desorption isotherms at -196 °C and (b) pore size distribution of B6-MJ(4)030 before and after C₆₀ masking. The ordinate axes were normalized to the mass of B6-MJ(4)030 alone.

pronounced characteristic of narrow micropore filling (<1 nm) [5]. In contrast, after C₆₀ masking (B6-MJ(4)030/C₆₀), almost no N₂ uptake at very low P/P_0 was observed. Meanwhile, the shape of adsorption and desorption isotherms in other P/P_0 regions was basically identical to that of B6-MJ(4)030. Consistently, micropores smaller than 1 nm disappeared from the pore size distribution of B6-MJ(4)030 after C₆₀ masking, whereas mesopores larger than 6 nm were almost unchanged (**Fig. 3(b)**). Therefore, we concluded that the C₆₀ masking method can also be applied to disordered and complex porous carbon materials.

Here, we considered the possibility that toluene could be trapped in the pores masked with C₆₀ molecules. As shown in **Fig. 3(b)**, the unmasked region (>6 nm) in the pore size distribution normalized by the weight of the porous carbon before and after C₆₀ masking was almost the same for B6-MJ(4)030. This suggests that even if such residual toluene exists, its amount is negligible. If a significant amount of toluene were trapped in the pores, the pore size distribution curve for B6-MJ(4)030/C₆₀ would be considerably lower than that for B6-MJ(4)030 due to the contribution of the weight of the trapped toluene.

Table 1 summarizes the pore structural parameters of three model porous carbon materials before and after C₆₀ masking. Here, the pore structural parameters are shown on a sample weight basis.

Next, we attempted to combine the molecular masking method with ¹²⁹Xe-NMR spectroscopy to selectively measure the unmasked pores in the porous carbon materials. **Fig. 4** shows the ¹²⁹Xe-NMR spectra of B6 and MJ(4)030 measured at -75 °C and 100 kPa of Xe adsorption temperature and pressure, respectively, along with the spectra of B6-MJ(4)030 before and after C₆₀ masking. The adsorbed Xe peak of B6 appeared at approximately 231 ppm, which was located downfield from that of MJ(4)030 ($\delta \approx 135$ ppm). The average pore size of B6 was smaller than that of MJ(4)030; therefore, the interac-

Table 1 Pore structural parameters estimated from the N₂ adsorption isotherms of three porous carbon materials at -196 °C before and after C₆₀ masking. The values are shown on a sample weight basis.

Sample	Specific surface area [m ² /g]	Total pore volume [cm ³ /g]
B6	780	0.25
MJ(4)030	560	2.89
B6-MJ(4)030	720	1.27
B6/C ₆₀	0	0
MJ(4)030/C ₆₀	90	0.53
B6-MJ(4)030/C ₆₀	50	0.21

tions between the adsorbed Xe molecules and the pore walls become stronger, leading to a more pronounced deformation of the electron cloud of the adsorbed Xe molecules and an enhanced deshielding effect [10, 13]. In the ¹²⁹Xe-NMR spectrum of B6-MJ(4)030, two distinct peaks attributed to the adsorbed Xe molecules were observed, one corresponding to that adsorbed on B6 (231 ppm) and the other to that adsorbed on MJ(4)030 (135 ppm). This result indicates that the Xe molecules were adsorbed in two different types of pores.

After C₆₀ masking, the peak at approximately 231 ppm disappeared completely, while the other peak at approximately 135 ppm remained essentially unchanged. These findings agree well with the N₂ adsorption and desorption isotherm results. For B6-MJ(4)030/C₆₀, the pores of B6 in the mixture were completely masked by C₆₀, whereas those of MJ(4)030 were unmasked. To verify the reproducibility of this phenomenon, we employed a different mesoporous carbon sample, MJ(4)010 (CNovel®; Toyo Tanso Co., Ltd., Japan), instead of MJ(4)030, and carried out the same experiments. MJ(4)010 was mixed with B6 to obtain the mixture B6-MJ(4)010, and C₆₀ masking of this material afforded B6-MJ(4)010/C₆₀. We then measured the ¹²⁹Xe-NMR spectra of B6-MJ(4)010 and B6-MJ(4)010/C₆₀. After C₆₀ masking, the ¹²⁹Xe-NMR peak of Xe adsorbed on B6 completely

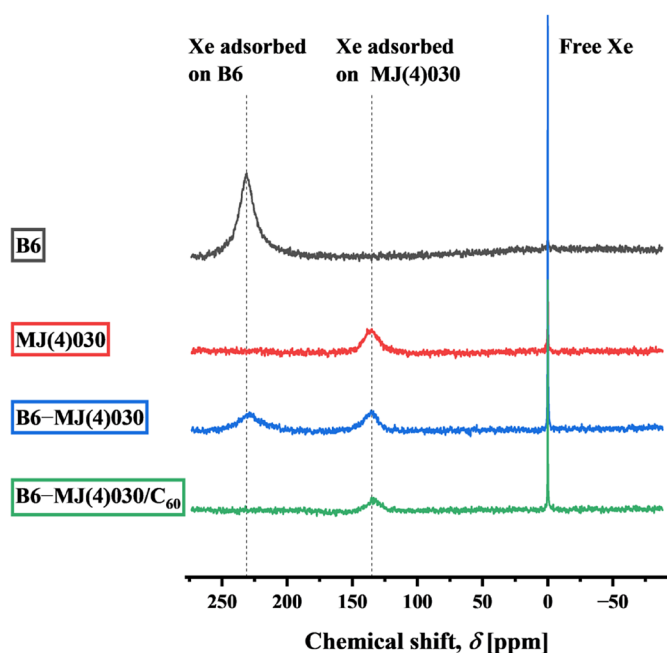


Fig. 4 ^{129}Xe -NMR spectra of B6, MJ(4)030, B6-MJ(4)030, and B6-MJ(4)030/C₆₀ measured at $-75\text{ }^{\circ}\text{C}$ under a Xe gas pressure of 100 kPa.

disappeared, while that of Xe adsorbed on MJ(4)010 was unaffected in the spectrum of B6-MJ(4)010/C₆₀, as shown in **Fig. S2**. These results indicate that the application of molecular masking enables the selective ^{129}Xe -NMR measurement of unmasked pores in disordered and complex porous carbon materials. Moreover, we recently demonstrated that adsorption-controlled ^{129}Xe -NMR spectroscopy is not only a practical technique for analyzing pore size but can also be applied as a general approach for analyzing pore shape and connectivity in a variety of porous materials [30]. Therefore, the present results highlight the potential of the combined method for individually evaluating the pore structures (*e.g.*, size, shape, and connectivity) of disordered and complex porous materials.

Currently, we are planning to explore the use of other masking agents to broaden the range of maskable pore sizes. These include fullerenes with different molecular sizes (*e.g.*, C₂₈, C₇₀, C₉₀, C₁₂₀) and three-dimensional aromatic molecules such as triptycene, three-dimensional non-aromatic molecules such as adamantane, and two-dimensional aromatic molecules such as pyrene. This will expand the applicability of the combined molecular masking and ^{129}Xe -NMR method to various disordered and complex porous materials.

4. Conclusion

This study proposed a pore-selective evaluation method for pore structure of disordered and complex porous carbon materials by combining molecular masking with ^{129}Xe -NMR spectroscopy. Vacuum impregnation of the carbon materials using a C₆₀-toluene solution enabled the complete masking of micropores smaller than 1 nm, while mesopores larger than 6 nm remained unmasked. This allowed us to

evaluate mesopores larger than 6 nm individually using ^{129}Xe -NMR spectroscopy. The ^{129}Xe -NMR spectrum of the C₆₀-masked model bimodal porous carbon (a mixture of microporous and mesoporous carbons) was consistent with that of the parent mesoporous carbon. The reliability of this method was also confirmed using a different mesoporous carbon material. In summary, this study demonstrated the potential of the proposed method, which combines molecular masking with ^{129}Xe -NMR spectroscopy, to selectively evaluate different pore structures in disordered and complex porous carbon materials.

Supporting Information

Mass ratio of porous carbon to C₆₀ used in this study. Pore structural parameters of samples using a mesoporous carbon material, MJ(4)010, estimated from N₂ adsorption isotherms at $-196\text{ }^{\circ}\text{C}$. N₂ adsorption and desorption isotherms of C₆₀ at $-196\text{ }^{\circ}\text{C}$. ^{129}Xe -NMR spectra of B6, MJ(4)010, B6-MJ(4)010, and B6-MJ(4)010/C₆₀ measured at $-75\text{ }^{\circ}\text{C}$ under a Xe gas pressure of 100 kPa.

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