Preparation and carbonization of glucose and pyromellitic dianhydride crosslinked polymers

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Supplementary Data

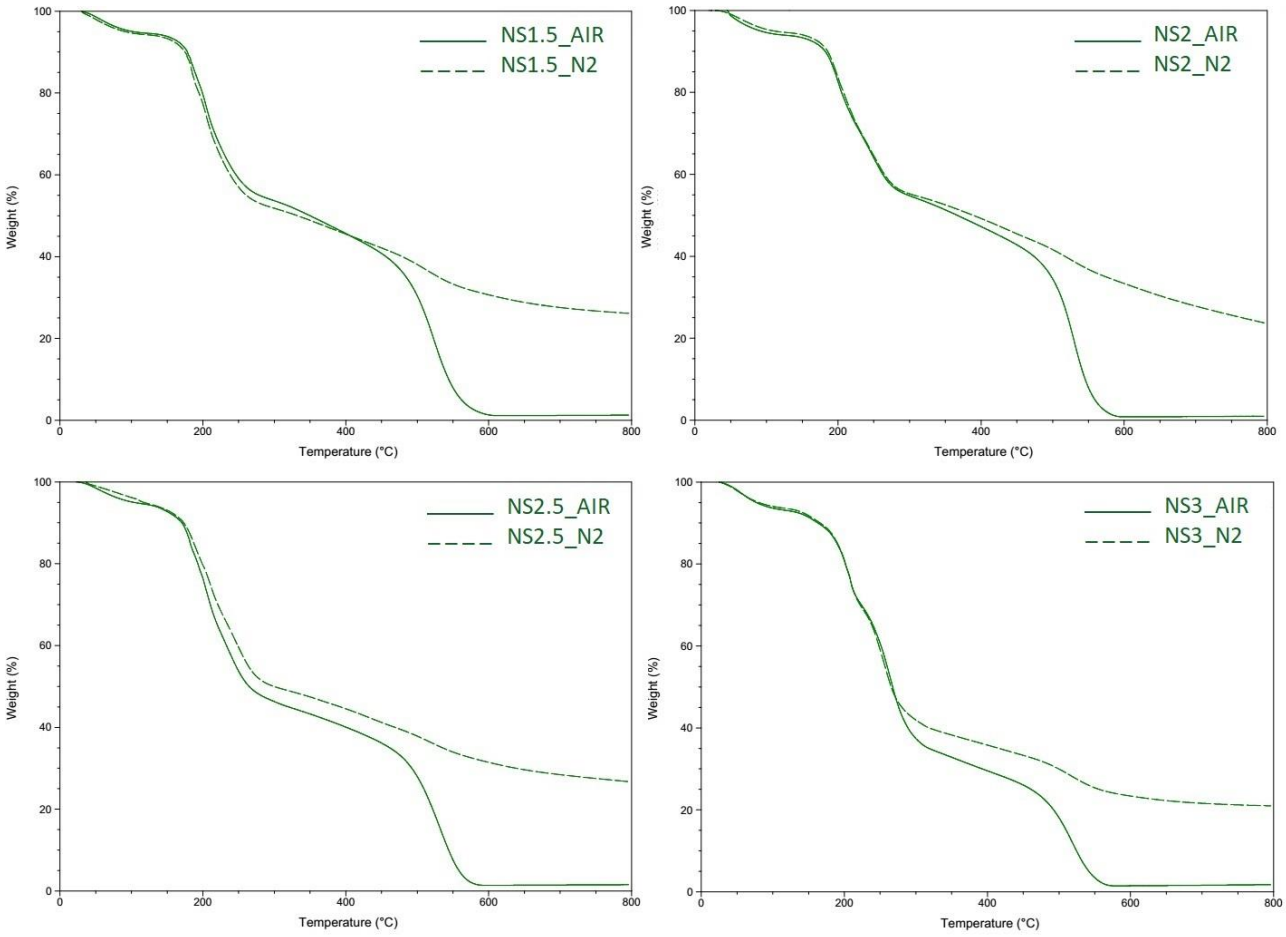

Figure S1. TG plots obtained under air or N_2 (solid or dotted lines, respectively) of NS1.5, NS2, NS2.5, NS3.

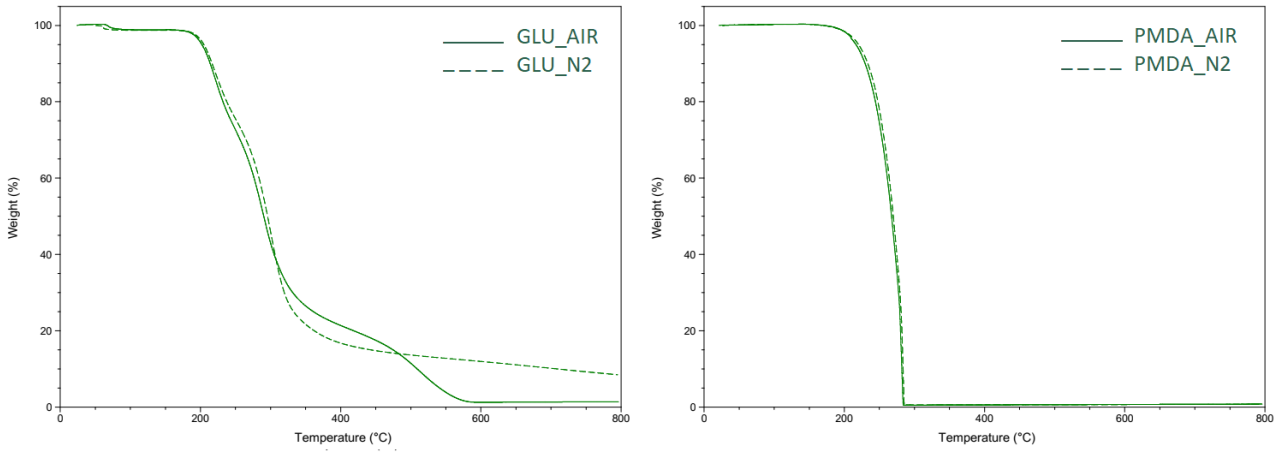

Figure S2. TG plots obtained under air or N_2 (solid or dotted lines, respectively) of GLU (left) and PMDA (right).

$S_{Langmuir}$, S_{BET}, S_{micro}, V_{micro} determination methods of pyrolyzed porous materials

1). Langmuir and BET-plots and calculations of the $S_{Langmuir}$ and S_{BET} [1]

$$\text{Langmuir equation: } \frac{P}{W} = \frac{1}{K\,W_m} + \frac{P}{W_M}$$

where P is the adsorbate equilibrium pressure, W and W_m are the adsorbed weight and monolayer weights, respectively. K is a constant.

A plot of P/W vs. P gives a straight line with $1/W_m$ as a slope for type I isotherms $0.05 \leq P/P_0 \leq 0.25$ region $S_{Langmuir}$ is calculated from the equation:

$$S_{Langmuir} = \frac{W_m\,\overline{N}\,A_{CS}}{\overline{M}}$$

where $\overline{N}$ is the Avogadro's number ($6{,}02 \times 10^{23}$), A_{CS} is the cross-sectional area of the adsorbate (16,2 Å^2 for N_2), $\overline{M}$ is the adsorbate molecular weight.

$$\text{BET Equation: } \frac{1}{W\left[\left(\frac{P_0}{P}\right)-1\right]} = \frac{1}{W_m\,C} + \frac{C-1}{W_m\,C}\left(\frac{P}{P_0}\right)$$

where W is the weight of gas adsorbed, P/P_0 is the relative pressure, W_m is the weight of adsorbate intended as a monolayer, C is the BET constant.

BET equation describes a linear plot, restricted to $0.05 \leq P/P_0 \leq 0.25$ region of the N_2 adsorption isotherm, of $1/[W(P_0/P)-1]$ vs. P/P_0 with $\frac{C-1}{W_m\,C}$ and $\frac{1}{W_m\,C}$ as slope and intercept, respectively.

The BET surface area is calculated from the slope and the intercept from the following equation:

$$S_{BET} = \frac{X_m\,\overline{N}\,A_{CS}}{M_v\,M}$$

Where $\overline{N}$ is the Avogadro's number, A_{CS} is the cross-sectional area of the adsorbate, M_v is the molar volume (22414 mL), and M is the sample mass.

2). Quantitative analysis of the S_{micro} and Vol $_{micro}$ by means of t-plot method (Carbon Black STSA thickness equation)

The method evaluates the thickness of N_2 as an adsorbate layer as a function of P/P_0 with $0.05 \leq P/P_0 \leq 0.35$, as compared to non-porous material. From the linear curve extrapolated to the P/P_0 axis, V_{micro} and S_{micro} are determined as the intercept and slope, respectively.

$$\text{Carbon Black STSA thickness equation (t)} = 0{,}88\,(P/P_0)^2 + 6{,}45\,(P/P_0) + 2{,}98$$

References:

[1] Lowell S., Shields J.E., Thomas M.A., Thommes M. (2004) Surface Area Analysis from the Langmuir and BET Theories. In: Characterization of Porous Solids and Powders: Surface Area, Pore Size and Density. Particle Technology Series, vol 16. Springer, Dordrecht. DOI: 10.1007/978-1-4020-2303-3_5

[2] ASTM D 6556-14 Standard Test Method for Carbon Black—Total and External Surface Area by Nitrogen Adsorption