

Letter to the editor

Glucose-derived carbon molecular sieve membrane: An inspiration from cooking



A B S T R A C T

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Polymeric precursor affects the structure of carbon membrane, and ultimately, its separating capability after pyrolysis. There, however, remains a large pool of unexplored polymer for low-cost, high-performing carbon membrane. Herein, we explore glucose, a simple sugar, as the precursor for carbon membrane. The idea originates from observing burnt char during cooking. The glucose layer on a supported ceramic is converted to carbon matrix at 180 °C and 50 bars in nitrogen. The carbon matrix exhibits size-sieving mechanism, with ideal H₂/N₂ and H₂/CH₄ selectivities of 11.6 and 9.9 respectively, and H₂ permeability of 8705 Barrer, exceeding the Robeson line.

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Pyrolyzing polymeric membrane rearranges the carbon backbone and releases volatile pendant groups to form carbon membrane with micro-porosity (2.5–5 Å) suitable to separate gases or organic solvent [1]. The turbostatic nature of the pyrolyzed carbon provides size-sieving mechanism with greater chemical and better thermal stability compared to polymeric membranes [2]. Thermosetting polymers, particularly polyimide-based, are traditionally favored as precursors for carbon membranes due to their ability to retain their structural integrity during pyrolysis [1]. Such polymers decompose at relatively high temperature (>400 °C), thus minimizes the probability of defects when the volatile groups leave the structure during pyrolyzing. The material space for carbon membrane's precursor, however, has expanded with the improved in science and fabrication techniques to include another classes of polymer; for example, conducting polymer [3,4], an unusual candidate for carbon membrane due to its limited solubility in organic solvent and low thermal resistivity, has been explored.

Here, we explore glucose, a simple sugar, as a carbon membrane precursor. We draw inspiration from the burnt char observed when overcooked sugar. Glucose is an important source of energy in biology and have a formula of H-(C=O)-(CHOH)₅-H where the five hydroxyl groups are attached along its six-carbon backbone. Glucose exists in either a thermodynamically unfavorable open-chain structure or a stable cyclic structure. The pyrolysis of glucose yields many possibilities including furans (5 membrane ring) and small oxygenates. We convert a glucose layer on a macro-porous ceramic (zirconia, See [Supplementary materials](#)) to carbonaceous material under highly energized environment. The high pressure (50 bars) and relatively low temperature (180 °C) suppress the volatile groups from decomposing and convert the glucose to carbonaceous structure. This highly energized environment also favors the formation of open-chain form which is paramount in closing the

defects by encouraging the connection of the carbon structure. The conversion of polymeric materials to carbon matrix occurs at around 180 °C for glucose, much lower than other polymeric system where temperature of >500 °C is needed. Thus far, to our best knowledge, there is no study on using carbon derived from sugar to make carbon membrane; only, the pyrolysis of sugar to yield carbon for anode material in lithium batteries [5] and characterization of carbon based on sugar [6] have been reported.

Fig. 1a shows the pure-gas permeability of the carbon matrixes with kinetic diameter of the gases. The carbon membrane derived from glucose (CM-Glu-0.133) shows a decreasing permeability trend with increasing kinetic diameter of the gases (i.e. gas permeability in the order of H₂ > CO₂ > O₂ > N₂ > CH₄), corresponding to a sieving mechanism where the smaller size molecules move faster across the membrane than the bigger ones. Molecular sieving is concluded to be the domineering mechanism, rather than Knudsen diffusion, by comparing the permeability of CO₂ with CH₄. The selectivity of CO₂/CH₄ (2.71) with CO₂ permeability of 2378 Barrer strongly suggests that size factor of the gas is largely important in its transportation across the glucose-derived membrane. Otherwise, a Knudsen diffusion-dominant matrix would give a permeation selectivity of 0.6 for CO₂/CH₄ instead [4]. Particularly, the high H₂ permeability suggests that such matrix can be used for processes that require purification of H₂. Plotting the ideal separating factors of H₂/N₂ (11.6) and H₂/CH₄ (9.9) with H₂ permeability (8705 Barrer) in Fig. 1b and c reveal that the data points are higher than the Robeson line, which is the upper boundary based on the current status of membranes (both polymeric and inorganic). This shows the potential of glucose-derived carbon membranes in competing with polyimide-derived carbon membranes. Examining the carbon membrane under electrons microscope shows a dense membrane of 29 μm on a supported porous YSZ ceramic (Fig. 2a).

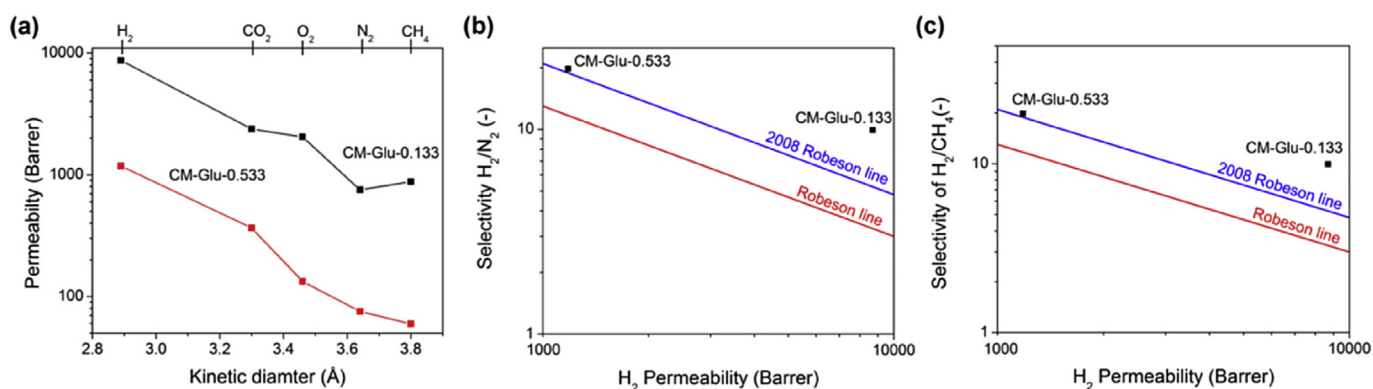


Fig. 1. (a) The dependency of gas permeability on kinetic diameter of penetrant molecules for the carbon membranes based on α -glucose as the precursor. The decrease in permeability with kinetic diameter of the gases tested for all the membranes shows that the dominant separation mechanism of the carbon matrix is molecular sieving, i.e. smaller size gas molecules penetrate through the membrane faster than the bigger size gas molecules. (b–c) The ideal selectivities achieved by the carbon membranes are plotted on a Robeson plot; (b) H₂/N₂ and (c) H₂/CH₄. The membranes perform better than most studies, i.e. higher than the Robeson upper boundary. The Robeson upper line was updated in 2008, indicated by the blue line. (A colour version of this figure can be viewed online.)

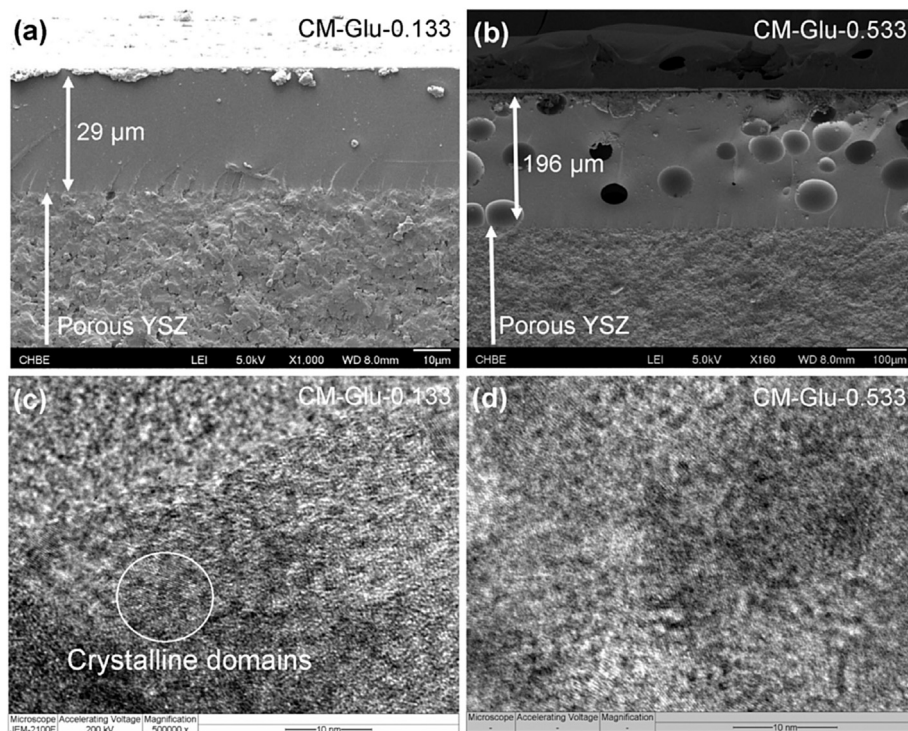


Fig. 2. (a–b) FESEM micrographs of the cross-section of the asymmetry carbon membrane: (a) CM-Glu-0.133, (b) CM-Glu-0.533. In each case, a dense carbon membrane on the ceramic support (YSZ) was obtained and the carbonized layer appears to adhere well to the ceramic. Using a less viscous coating solution (0.133 g of glucose per ml of water) results in a thinner carbon membrane (29 μm). However, pockets of circular spheres, which appears to be due to the air bubbles, are observed in the thicker carbon membrane based on higher viscous coating solution. (c–d) TEM micrographs of carbon matrix: (a) CM-Glu-0.133, (b) CM-Glu-0.533. Crystalline domains appear in each of the sample with lattice spacing ~ 0.255 nm for the CM-Glu-0.133 and CM-Glu-0.533.

No apparent crack line between the dense membrane and the ceramic suggests good affinity between the two materials. In addition, the thermal expansion coefficient, α , of these materials should be in the same order of magnitude ($\alpha_{\text{YSZ}} = 7 \times 10^{-6} \text{ K}^{-1}$) [7].

We increase the thickness of the carbon membrane by using a more concentrated glucose solution, CM-Glu-0.533 (4-fold increase in concentration from 0.133 g of glucose/ml of water compared to 0.533 g of glucose/ml of water). This results in a dense carbon membrane of 196 μm as seen in Fig. 2b. This matrix exhibits similar trend of decreasing permeability with gas diameter to the thinner membrane (CM-Glu-0.133), albeit at an-order-magnitude lower permeability. Coupled with the ideal selectivity of 6.13 for CO₂/CH₄, we

concluded that molecular sieving is the dominant mechanism for CM-Glu-0.533. On the whole, the performance of this membrane still lies above the Robeson line (Fig. 1b and c) for the selected gas pairs of H₂/N₂ and H₂/CH₄. CM-Glu-0.533 exhibits ideal separating factors of 15.6 for H₂/N₂ and of 19.8 for H₂/CH₄ with H₂ permeability of 1182 Barrer. The higher viscosity coating solution (4 cP for 0.533 g/ml vs 1 cP for 0.133 g/ml) resulting in lower permeance due to higher mass transport resistance is also observed in similar studies [8]. Interestingly, we observe voids in the selective layer of CM-Glu-0.533 (Fig. 2b), which is attributed to the air bubbles trapped during pyrolysis. These air bubbles originate from the pores of the YSZ substrate and diffuse into the glucose layer during

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