

## POROUS ACTIVATED BINDERLESS PELLETS FOR ELECTROCHEMICAL OXYGEN REDUCTION AND EVOLUTION REACTIONS

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### Introduction

The oxygen evolution reaction (OER) and the oxygen reduction reaction (ORR) are important electrochemical reactions for sustainable energy devices such as water splitting technology and fuel cells.[1] Traditionally, noble metal catalysts (Pt, Ru, Ir) or transition metals and metal oxides (Ni, Co) have been considered as the best electrocatalysts due to its high activity.[2, 3] However, the high cost, scarcity, and poor long-term stability of these noble metal-based catalysts significantly hinder their large-scale applications.[4, 5] In this sense, metal-free carbon catalysts have attracted much attention in these energy conversion and storage technologies because of its good electronic conductivity and high activity, in addition to their low-cost and wide availability.[6] Here, we present a simple strategy to produce porous activated carbon materials in the form of pellet derived from the hydrochar prepared via hydrothermal carbonization of glucose solution. The hydrochar obtained was then pressed in the form of pellet and subjected to physical activation with CO<sub>2</sub> or mixed with KHCO<sub>3</sub> to increase the textural properties of the carbon discs. Since the materials obtained by this synthesis procedure are mainly microporous, a template strategy was also employed to produce mesoporous carbon materials by using SBA-15 as the template. In this sense, a comparison of the role of the textural properties was examined upon the catalytic activity of the carbon materials.

### Materials and Methods

An aqueous solution of glucose was placed in a stainless steel autoclave and heat-treated at 220 °C for 5 hours. After the hydrothermal treatment, the solid product was filtered, washed thoroughly with abundant water and dried.

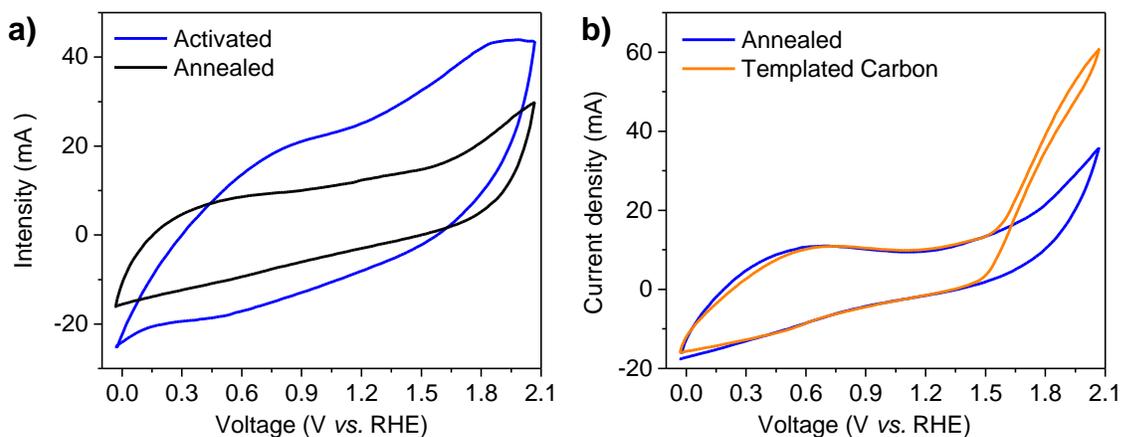
For the physical activation process, first the solid hydrochar powder was pressed to conform a pellet. Then, the hydrochar pellets were placed in a tube furnace and heat-treated up to a desired temperature in an argon atmosphere. Once the activation temperature was reached, the argon flow was switched to CO<sub>2</sub> and maintained during the activation time. Afterwards, the gas flow was switched again to an inert atmosphere.

In case of the chemical activation, the hydrochar was thoroughly mixed with KHCO<sub>3</sub> at KHCO<sub>3</sub>/hydrochar weight ratio of 1 and 2 in an agate mortar. Then, the resulting mixtures were compacted to obtain pellets, which were placed in a tube furnace and heat-treated to the desired activation temperature under an argon gas flow and held at the activation temperature for 1 h. The resulting carbon pellets were washed several times with 15 wt% HCl to remove any inorganic salts, and then with abundant distilled water. No stirring was used during the washing step. Finally, the carbons were dried in an oven several hours.

Finally, in the case of the templated porous carbon the synthesis was as follows. To a tube was added 5.46 g of water, 1 g of SBA-15 (synthesized according to a previously reported procedure) and 1.20 g glucose. The tube was then sonicated and stirred until all the particles were evenly dispersed. The mixture was heated at 100 °C and then at 250 °C for 6 h each. The process was repeated until the pore volume was reduced to approximately zero. Then, the resulting composite was compacted to obtain pellets. The complete pyrolysis of carbon-pellets was carried out under argon atmosphere at 1000 °C in a tube furnace. Finally, the silica template was removed by etching with NaOH 2M several days and washing the resulting HTCs with abundant distilled water. For electrochemical measurement, a conventional three-electrode set-up cell was used with Pt wire as the counter electrode and mercury/mercury oxide electrode (Hg/HgO) in alkaline media as the reference electrode. The working electrode (in the form of disc) is mounted by PEEK electrode holder.

## Results and Discussion

Linear sweep voltammetry and cyclic voltammetry at high anodic potential were carried to measure the OER activity of HTC disc. The higher oxidation current comes from not only oxygen evolution by oxidizing water, but also carbon oxidation from the carbon pellet. Thus, we also considered the stability of the porous pellets at OER by applying a current density to observe the change of the overpotential at highly oxidative conditions. As can be observed from Figure 1a and 1b, the materials exhibited an enhanced on the electrocatalytic activity.



**Figure 1.** Comparison of the cyclic voltammeteries in 0.1 M KOH at 5 mV s<sup>-1</sup> for a) physically activated carbon and carbonized pellets and b) templated porous carbon and carbonized pellets.

## Conclusions

To conclude, we have presented the synthesis of porous activated carbon materials in the form of a pellet without the employ of any binder. Hydrothermal carbonization was first used to convert low cost glucose into a hydrochar that was pressed to obtain a pellet. Three different strategies were employed to modify the textural properties of the materials: a) physical activation, b) chemical activation and c) Nano-templating. The materials were tested as electrocatalysts for OER in basic electrolyte. An enhancement on the current density was observed due to the modification

of the textural properties. In addition, a comparison of the role of the textural properties was examined upon the catalytic activity of the different carbon materials.

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