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Direct Conversion of Rice Husks to Nanostructured SiC/C for CO₂ Photoreduction

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Abstract

A one-step and template-free synthesis of a SiC nanowires/C (SiC-NW/C) composite from rice husks (RHs) is realized via a molten salt-assisted electrochemical method. The process integrates simultaneously carbonization, electrodeoxidation, nanostructuring and self-purification for converting RHs to SiC-NW/C hybrid that is assembled from SiC NWs embedded in porous N-doped graphitic carbon with strong coupling. The SiC-NW/C nanostructure enables efficient CO₂ adsorption and fast separation and transfer of charge carriers. Benefiting from the structural and compositional merits, the SiC-NW/C composite shows superior activity for photoreduction of CO₂ to CO, in the absence of any additional cocatalysts or sacrificial agents. The process proposed herein might help to bridge a closed-loop carbon cycle in the whole production-utilization of biomass.

Keywords: molten salt electrolysis; biomass utilization; SiC; CO₂ reduction; rice husks.

Primary production is a photo/chemo-synthesis process to convert CO₂, H₂O and inorganic nutrients into massive biomass, which plays an essential role in carbon neutrality.^[1] Biomass is therefore composed of organic mass and inorganic components.^[2] In recent years, carbon neutrality tends to be compromised by traditional combustion-utilization of biomass with huge carbon emission.^[3-4] Exploring new technologies to guarantee carbon balance in the production-utilization cycle of biomass is of prime importance.

Rice husks (RHs), a typical agricultural waste, contain 75~85 wt% of organic mass, 15~20 wt% of silica and trace amounts of inorganic elements (e.g., K, S, Mg, Zn, and P).^[5-6] RHs have been utilized as a resource to produce silicon- or carbon-based materials.^[7-8] For example, RHs can be pre-treated by thermal annealing to remove organic mass and alkaline/acid leaching to eliminate metallic impurities, generating a silica product after the separation process.^[9-10] Silicon will then be produced by reducing the obtained silica under harsh conditions coupled with post acid etching treatments to remove impurities.^[11-14] Alternatively, carbonaceous materials can be harvested through high-temperature pyrolysis of RHs followed by multistep wet chemical treatments for purification.^[15-16]

Although these strategies have achieved the resource utilization of RHs, they involve several main drawbacks. First, simultaneous conversion of organic mass and biosilica of RHs to functional moieties of targeted materials remains to be realized.^[5, 8] Besides, the multistep synthetic methods require tedious processes to separate and purify the products.^[5] Meanwhile, formation of the useless by-products (e.g., ash and volatile liquid) will impede continuous production of the materials or even poison them to loss catalytic activity.^[6, 17] Moreover, preparation of hybrid nanostructures from RHs is seldom achieved via conventional high-temperature-processed methods. Therefore, engineering RHs to value-added materials, especially the candidates enabling renewable fuel generation, through a facile and efficient process is still highly desirable, yet very challenging.

Herein, we present a one-pot molten salt-assisted electrochemical synthesis of a SiC-nanowires/C (SiC-NW/C) composite from RHs as an efficient photocatalyst for CO₂ reduction, which promises a sustainable carbon cycle. As illustrated in **Figure 1a**, RHs are electrochemically converted to SiC-NW/C and CO₂ in molten salts of NaCl-CaCl₂. The released CO₂ gas can supply primary production to form biomass or be photoreduced to CO by the as-derived SiC-NW/C (Figure S1, Supporting Information). In return, the generated CO fuel can power the molten salt electrolyzer. Therefore, the conversion of RHs to SiC-NW/C bridges in theory a closed-loop carbon cycle. Under simulated sunlight irradiation, the SiC-NW/C photocatalyst shows high CO₂-to-CO reduction activity without assistance of any cocatalysts or sacrificial agents.^[18]

Electrolysis in molten NaCl-CaCl₂ at 800°C is performed at a voltage of 2.6 V between the RHs cathode and graphite anode for 2 h, converting RHs to cathodic SiC-NW/C and anodic CO₂ (see Supporting Information for the details). As illustrated in Figure 1b, this molten salt method has obvious advantages of simultaneous carbonization, electrodeoxidation, nanostructuring and purification for transforming RHs to the SiC-NW/C composite in a one-step and template-free manner. Specifically, the pyrolysis of organic substances, the electrochemical deoxidation of biosilica and the formation of SiC NWs occur concurrently in the RHs cathode. Molten salt is a water-free electrolyte in liquid state, consisting of free ionic cations and anions. In particular, CaCl₂-based molten salt possesses a high solubility for metal oxides, which means that metal oxides can be easily ionized to metal cations and O²⁻ anions, thus enabling the free diffusion of O²⁻ in molten salts. Therefore, O²⁻ anions can be released from the RHs cathode during the electrolysis of biosilica, and transfer to the anode to be discharged into gaseous CO₂. Moreover, the inorganic impurities (e.g., K, S, Mg, Zn, and P) will dissolve into the molten salts spontaneously, fulfilling an in-situ purification for the SiC-NW/C product.

The morphology of SiC-NW/C is studied by field-emission scanning electron microscopy (FESEM) and transition electron microscopy (TEM). The FESEM images of SiC-NW/C show that

one-dimensional (1D) NWs with diameter of 10-30 nm and length up to several micrometers are homogeneously distributed (**Figure 2a,b**), which is far different from the bulky structure of the RHs precursor (Figure S2, Supporting Information). These NWs are revealed to be SiC by elemental mapping analysis (Figure S3, Supporting Information). A closer FESEM examination displays the intimate adhesion of SiC NWs on the carbon matrix (Figure 2c), indicating the configuration of SiC NWs inserted into the carbon support. The TEM images expose the interconnected structure between SiC NWs and carbon in SiC-NW/C (Figure 2d,e and Figure S4, Supporting Information). From the high-resolution TEM (HRTEM) images (Figure 2f-h), the interfaces between SiC NW and carbon are observed clearly. The distinct lattice fringes with an interlayer spacing of 0.25 nm correspond to (111) crystal plane of cubic β -SiC (Figure 2g). Moreover, beside SiC NW, some carbon domains with an interlayer distance of 0.34 nm can also be discerned (Figure 2h), suggesting the partially graphitized nature of the carbon. These results demonstrate the distinctive interconnected structure of SiC-NW/C constructed by a carbon matrix and embedded SiC NWs (Figure 2i).

Powder X-ray diffraction (XRD) pattern of the SiC-NW/C product gives four diffraction peaks at 35.6, 41.4, 60.0 and 71.8° (**Figure 3a**), assigning to (111), (200), (220) and (311) crystal planes of cubic β -SiC (JCPDS card no.: 29-1129), respectively. The shoulder peak with a 2θ value of 33° is attributed to stacking faults in β -SiC.^[18] The weak and broaden peaks at around 24 and 44° belong to (002) and (100) planes of graphitic carbon with low crystallinity,^[19] corresponding to the reflections of graphitic-type lattice and disorder stacking of micro-graphite, respectively. The existence of both SiC and C in SiC-NW/C is further verified by Raman analysis (Figure 3b). The two bands at 785 and 956 cm^{-1} represent the transverse optic (TO) and longitudinal optic (LO) phonon modes of β -SiC,^[20] while the small peak at 1510 cm^{-1} reflects the two-phonon or combinational bands of β -SiC.^[21] Besides, the strong bands at 1398 and 1576 cm^{-1} indicate the disordered (D band) and graphitic (G band) structures of carbon.^[22] After removing the carbon in

SiC-NW/C by calcination in air, the produced SiC-NW sample (Figure S5, Supporting Information) shows no carbon peaks in its XRD pattern and Raman spectrum (Figure 3a,b). The contents of C and SiO₂ in RHs are determined to be 20.5 and 14.7 wt% (Figure S6a, Table S1, Supporting Information), which means the theoretical molar ratio of SiC to C in SiC-NW/C should be 1:6 if RHs are converted completely (Figure S6b, Supporting Information). Calculations from the thermogravimetric analysis (TGA) results of SiC-NW/C in air indicate that the molar ratio of SiC/C in SiC-NW/C is 1:5.8 (Figure S7, Supporting Information), which is very close to the theoretical value. All these observations highlight that the molten salt-assisted electrochemical approach renders thorough conversion of RHs to SiC-NW/C with high yields.

The contents of impurity elements in pristine RHs and SiC-NW/C product are determined by inductively coupled plasma optical emission spectrometry (ICP-OES). As shown in Table S2 (Supporting Information), the amounts of the impurities in SiC-NW/C are much lower than those in RHs. After the molten salt treatment, the removal rates of K, S, Mg, Zn and P elements are 99, 95, 85, 80 and 77% (Table S2, Supporting Information), respectively. This result suggests that the molten salt can efficiently absorb the detrimental impurities, thus ensuring the self-purification ability of the preparation technique.

The chemical states of elements in SiC-NW/C are investigated by X-ray photoelectron spectroscopy (XPS). Besides Si and C elements, N is also detected in the survey spectrum with a content of 0.8 wt% (Figure S8 and Table S3, Supporting Information), which is consistent with the result of EDX test (Figure S9, Supporting Information). In the XPS spectra of N 1s and C 1s (Figure 3c,d), the N signal is only detected in SiC-NW/C, and correspondingly the peak assigned to C-N bond (e.g., 286.0 eV) is identified only in SiC-NW/C,^[23] but not in pure SiC-NW. Such results prove that the N element in RHs is doped into the carbon of SiC-NW/C, which is further verified by the absence of N signal in the EDX spectrum of SiC-NW (Figure S9, Supporting Information). In addition, the EDX results also show that SiC-NW possesses less O than Si-NW/C (Figure S9,

Supporting Information), suggesting SiC-NW is not oxidized during the removal of C from Si-NW/C. In the Si 2p XPS spectra (Figure 3e), the Si-C peak in SiC-NW/C is positively shifted by 0.38 eV compared to that in SiC-NW, further reflecting the strong interaction between SiC and C.^[23] The small Si-O peak at 101.8 eV is due to the minor oxidation of surface SiC. Meanwhile, the Si-C peak in the C 1s XPS spectrum of SiC-NW/C is about 0.2 eV higher than that of SiC-NW. The energy increase in the Si-C bond of SiC-NW/C implies that electrons tend to transfer from SiC to C,^[24] which thus can restrain recombination of charge carriers for SiC-NW/C during photocatalysis.^[25] Importantly, N-doping can ameliorate the electronic structure and enhance the conductivity of graphitic carbon, thus favoring the quick electron transfer from SiC-NW to C in SiC-NW/C.^[26-27] Furthermore, the N-doped graphitic carbon of SiC-NW/C is proved to be of porous structures given the fact that the composite has a specific surface area three times larger than SiC-NW (Figure 3f, Figure S10 and Table S4, Supporting Information). The highly porous feature of SiC-NW/C affords the material a maximum CO₂ adsorption of about 41.5 cm³ g⁻¹ at 273 K (Figure S11, Supporting Information).

To further explore its promise for CO₂ photoreduction, the optical harvesting ability and band structure of the SiC-NW/C hybrid are determined. It is revealed that SiC-NW has broad optical response from UV to visible region and a band gap of 2.41 eV (Figure S12a,b, Supporting Information). By further combining the results of Mott-Schottky curve and XPS valance band (VB) spectrum (Figure S12c,d, Supporting Information), the conduction band (CB) and VB positions of SiC are defined to be -1.31 and 1.1 eV (vs. normal hydrogen electrode, NHE, pH = 7), respectively. Therefore, the SiC-NW/C composite has sufficient redox potentials for CO₂ reduction reactions.^[28]

Photocatalytic CO₂ reduction performance of the SiC-NW/C nanocomposite is evaluated in a typical gaseous CO₂/water system under simulated sunlight irradiation,^[29] without using any cocatalysts or sacrificial agents. CO is detected as the main reduction product coupled with small yields of CH₄ and H₂ (**Figure 4a**), suggesting the high selectivity of SiC-NW/C for CO₂

photofixation. Besides, the oxidation product of O_2 is also observed in the system (Figure S13, Supporting Information). The reason that CO is the primary product may be ascribed to the lower CB potential of SiC-NW. Indeed, this potential is still thermodynamically sufficient to reduce CO_2 to CH_4 ; however, the formation of CH_4 typically proceeds via a series of elementary steps with CO as the intermediate, involving transfer of one electron in most of the steps.^[30] When the reduction potential of the excited electron is lower, the reaction rates of the elementary steps become kinetically slower.^[31] Therefore, the successive reaction steps after CO formation cannot proceed fast enough before CO desorbs from the surface of the photocatalyst, thus leading to formation of CO as the main product. After reaction for 3 h, the SiC-NW/C photocatalyst exhibits a total CO yield of $17.6 \mu\text{mol g}^{-1}$ and O_2 yield of $13.1 \mu\text{mol g}^{-1}$ (Figure S13, Supporting Information), delivering an average CO-producing rate of $5.87 \mu\text{mol g}^{-1} \text{h}^{-1}$ and O_2 evolution rate of $4.37 \mu\text{mol g}^{-1} \text{h}^{-1}$. This CO_2 reduction activity of SiC-NW/C is comparable to that of reported works (Table S5, Supporting Information). In comparison, the pure SiC-NW manifests much inferior activity with a CO generation rate of only $1.77 \mu\text{mol g}^{-1} \text{h}^{-1}$ (Figure 4b and Figure S13, Supporting Information), which indicates the synergy of SiC-NW and C in improving the photocatalytic activity of the Si-NW/C hybrid. The control experiments show that the CO_2 reduction reaction only occurs when SiC-NW/C, CO_2 and light irradiation coexist in the system (Figure 4c). Using $^{13}\text{CO}_2$ as the gas source (Figure S14, Supporting Information), ^{13}CO ($m/z = 29.1$) is detected as the dominant product (Figure S15, Supporting Information). In addition, the gas chromatograph spectra present evident time-dependent increases in the products generation (Figure S16, Supporting Information). All these findings demonstrate that the SiC-NW/C hybrid can efficiently catalyze the photoreduction of CO_2 to CO. The stability tests reveal that the SiC-NW/C photocatalyst preserves its main activity after repeatedly operating the CO_2 reduction reaction for three cycles (Figure S17, Supporting Information), suggesting its high stability and good reusability. Moreover, the XRD, XPS, FESEM

and TEM tests of the used sample further support the high stability of SiC-NW/C in the CO₂ reduction system (Figures S18 and S19, Supporting Information).

To elucidate the enhanced CO₂ reduction activity of SiC-NW/C, photoluminescence (PL) characterizations are conducted. The SiC-NW/C nanostructure presents much weaker PL emission than SiC-NW (Figure 4d), indicating restrained electron-hole recombination in the hybrid.^[32] Moreover, photoelectrochemical tests reveal that SiC-NW/C exhibits much higher transient photocurrent than SiC-NW (Figure 4e). The apparent PL emission decline and enriched photocurrent response indicate the accelerated separation and migration of charge carriers in the SiC-NW/C nanostructure,^[33] which enhances the CO₂ conversion efficiency. At last, the possible photocatalytic CO₂ reduction process over SiC-NW/C is proposed (Figure 4f). In the composite, the 1D SiC-NW with appropriate band structure absorb the photos to generate electrons upon light irradiation. Due to the strong coupling between SiC-NW and the N-doped graphitic carbon with excellent charge mobility, the excited electrons can quickly migrate to the porous carbon for reducing the adsorbed CO₂ molecules to CO. That is, the unique structural and compositional benefits of the nanostructured SiC-NW/C hybrid contribute together to the greatly enhanced CO₂ reduction performance.

In summary, we demonstrate a one-step molten salt-assisted electrochemical synthesis strategy for value-added utilization of plant biomass. By using CaCl₂-NaCl molten salt as the electrolyte, solid rice husks are directly converted to SiC-nanowire/C hybrid. This nanostructured composite shows much enhanced activity for CO₂ photoreduction to CO without employing any cocatalyst or sacrificial agents. The conversion of rice husks to SiC-nanowire/C promises a closed-loop CO₂ utilization by combining the molten salt treatment, photocatalysis and primary production in the biosphere. The molten salt system has the self-purification ability to dissolve detrimental elements, thus warranting a smart process for biomass utilization free of any refinement steps. The developed process might also bridge a balanced carbon cycle in the production-utilization of biomass.

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Figures and Captions

Figure 1. Schematic illustrations of (a) closed-loop carbon cycle and (b) reaction process of molten-salt assisted electrochemical conversion of RHs to SiC-NW/C for CO₂ photoreduction.

Figure 2. (a-c) FESEM images, (d,e) TEM images, (f-h) HRTEM images and (i) schematic illustration of SiC-NW/C nanostructure. Panels (f), (g) and (h) are based on regions 1, 2 and 3 in panel (e), respectively.

Figure 3. (a) XRD patterns, (b) Raman spectra, (c-e) XPS spectra, and (f) N₂ sorption isotherms of SiC-NW/C and SiC-NW.

Figure 4. (a) CO₂ photoreduction performance of SiC-NW/C. (b) Time-dependent CO yields of SiC-NW/C and SiC-NW. (c) Activity of reaction system under various conditions. (d) PL spectra and (e) photocurrent response of SiC-NW/C and SiC-NW. (f) Illustration of CO₂ photoreduction process over SiC-NW/C.

for Table of Content Entry

A one-step and template-free molten salt-assisted electrochemical method efficiently converts rice husks to SiC nanowires/C for enhanced CO₂ photoreduction, which in theory bridges a closed-loop carbon cycle in the whole production-utilization of biomass. This nanostructured SiC nanowires/C composite shows much enhanced activity for CO₂ photoreduction to CO without employing any cocatalyst or sacrificial agents.