nanomaterials

MDPI

Supplementary Materials

Derivation of Luminescent Mesoporous Silicon Nanocrystals from Biomass Rice Husks by Facile Magnesiothermic Reduction

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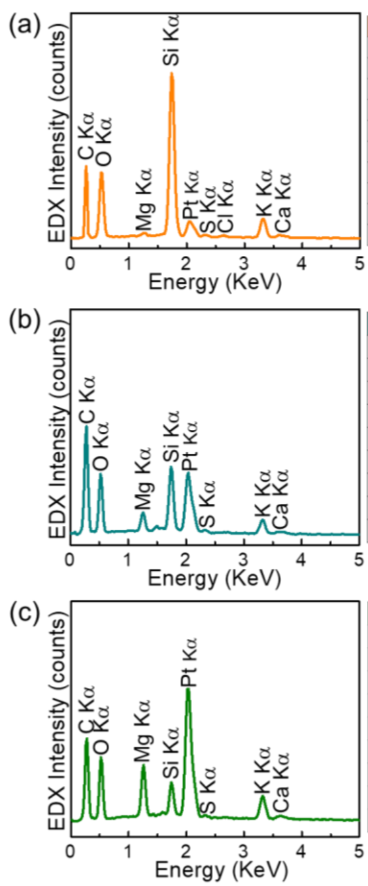
• Chemical Composition of Rice Husk Ashes



(a)

Element	Weight (%)
C	13.74
Si	24.62
O	39.89
K	6.26
Cl	2.48
Ca	2.56
Mg	3.65
S	2.12
Pt	4.68
Total	100

(b)

Element	Weight (%)
C	52.28
Si	5.95
O	27.56
K	2.64
Ca	1.07
Mg	1.79
S	0.57
Pt	8.14
Total	100

(c)

Element	Weight (%)
C	50.39
Si	2.46
O	26.35
K	4.84
Mg	6.21
Ca	1.93
S	0.34
Pt	7.48
Total	100

Figure S1. EDX spectra of (**a**) S-RH, (**b**) R-RH, and (**c**) B-RH ashes. Note that Pt in each raw source material arose from the conductive coating of Pt for better focusing and imaging during SEM and EDX measurements.

- **Comparison of Various Methods for Silicon Production**

Table S1. Summary of silicon synthesized from various resources through several experimental methods.

Method	Need for High-Vacuum Facility?	Precursor Materials	Hazardousness	Thermal Budget (Temperature, Power, etc.)
Laser Ablation	Yes	Silicon Bulks or Powders (Solid Phase)	No	High Power Excimer Laser (>a few J/cm^2)
Plasma Process	Yes	SiH_4, $SiCl_4$, etc. (Gas Phase)	Yes	RF Power (a few tens to hundreds W)
Pulsed Laser Deposition	Yes	Si or SiO_2 Ceramic Targets (Solid Phase)	No	High Power Excimer Laser (>a few J/cm^2)
Chemical Vapor Deposition	Yes	SiH_4, $SiCl_4$, etc. (Gas Phase)	Yes	High Temperature (~1000 °C)
Thermal Annealing	Yes	Siliceous Sources (Solid or Gas Phases)	Dependent on Precursors	Moderate Temperature (~500 °C)
Chemical Doping	Yes	Siliceous Sources (Solid and Liquid Phases)	Dependent on Precursors	Medium Temperature (700–800 °C)
Electrochemical Etching	No	Silicon Bulks or Powders (Solid Phase)	No	High Current Density (a few A/cm^2 for 1 min)
Molten-Salt Process	No	Every Siliceous Source and $CaCl_2$ (Solid Phase)	No	Medium–High Temperature (900–1000 °C)
Magnesiothermic Reduction	No	Every Siliceous Source and Mg (Solid Phase)	No	Medium Temperature (700–800 °C)

Table S2. Summary of silicon synthesized from various biomass resources through several experimental methods.

Study	Resources	Synthesis Method	Summary of Process Steps	Results
Z. Favors et al. [1]	Beach Sand	Magnesiothermic Reduction	· Calcination of sand at 900 °C in air · HCl, HF, and NaOH leaching · Ultrasonication for 1 h · NaCl was mixed with SiO_2 (10:1 wt.%); then, the mixture was ultrasonicated for 4 h · SiO_2:NaCl (1:0.9 wt.%) with Mg powder · Transferred to swagelok-type reactors · Annealed at 700 °C for 6 h in Ar-filled glovebox · HCl (5 M) and HF (10%) acid etching	· Porous network of interconnected crystalline silicon nanoparticles with high specific surface area of 323 m^2 g^{-1}
M. Sakamoto et al. [2]	Rice Husks	Pulsed Laser Melting	· HCl acid leaching · RH annealed at 700 °C for 4 h by flowing O_2 in furnace · SiO_2 mixed with Mg powder · Annealed at 650 °C for 2 h under H_2/Ar gas · HCl:Ethanol:H_2O (1.5:10:5) etching · Nd:YAG laser (λ = 532 nm) was used as an energy source for melting processes · Repetition rate: 10 Hz · Irradiation time: 20 min · Laser fluence: 50, 150, and 250 mJ/cm^2	· Nanocoral Si spheroidal structure with a particle size of ~200 nm · When increasing the laser fluence (50 to 250 mJ/cm^2 pulse), the specific surface area of the Si nanoparticle was decreased (57.9 to 20.7 m^2 g^{-1}).
J.-H. Choi et al. [3]	Rice Husks	Molten-Salt Process	· Acid leaching and thermal process of RH · RH-SiO_2 was mixed with NiO (20:1 at.%) in ethanol · Polyvinyl alcohol and zinc stearate were added as binders · Powders pressed at 100 bar in cylindrical mold · Sintering at 1200 °C in air for 5 h · Annealing of $CaCl_2$ at 850 °C in Ar atmosphere · RH-SiO_2 + NiO pellet was wrapped in nickel mesh · Electrodeoxidation was performed at 2.7–2.9 V for 0–10 h · HCl (0.1 M) and HF (2%) etching	· Crystalline Si nanowires with diameter of ~300 nm and length of ~1 μm · Si nanowires had excellent cycling and power performance in LIB anodes

A. Su et al. [4]	Corn Leaves	Aluminothermic Reduction	· Annealed at 650 °C in air for 3 h · HCl (1 M) leaching for 12 h · SiO_2 was mixed with Al powder and $AlCl_3$ powder · Annealed at 250 °C for 12 h under Ar atmosphere	· Crystalline silicon nanoparticles with specific surface area of 64 m^2g^{-1} · Si nanoparticles exhibited excellent long-term cycling and high rate capability in LIB anodes
S. Praneetha et al. [5]	Rice Husks	Microwave-Assisted Metallothermic Reduction	· HCl acid leaching · Powder transferred to swagelok-type reactors · Microwave solid-state process at 650 °C for 30 min · Operated frequency at 2.45 GHz · HCl:Ethanol:H_2O (19.3:172.6:28.3 mL) etching · Stirring for 6 h · Centrifuged and washed with water and ethanol, and dried in vacuum oven	· Interconnected nanoporous wall structure of Si with a wall thickness of ~23 nm and a pore diameter of 50–80 nm · It was used as a suitable material for LIB anodes.
Present Work	Rice Husks (3 Types of RHs)	Magnesiothermic Reduction	· Carbonized at 500 °C for 2 h in air · HCl leaching · Incineration at 700 °C for 2 h in air · SiO_2 mixed with Mg powder · Incineration at 700 °C for 2 h in Ar atmosphere · HCl (HCl:H_2O:EtOH = 0.66:4.72:8.88 molar ratio) etching for 10 h · HF acid etching · DI washing and drying	· Crystalline nature of spherical Si nanoparticles with average particle sizes of 15–50 nm · High surface area of 265.6 m^2 g^{-1} and high porosity · Light absorption near the UV region · Blue, green, and yellow emissions · The Si nanocrystals possess both high porosity and high luminescence/absorbance, which is indicative of great potential for highly efficient photocatalytic applications

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