

Electronic Supplementary Material (ESI)

STEP Organic Synthesis: An Efficient Solar, Electrochemical Process for the Synthesis of Benzoic Acid

Yanji Zhu, Baohui Wang*, Xuelin Liu, Huaiyuan Wang, Hongjun Wu and Stuart Licht*

The ESI contains:

Outdoor and indoor photocurrent configurations.

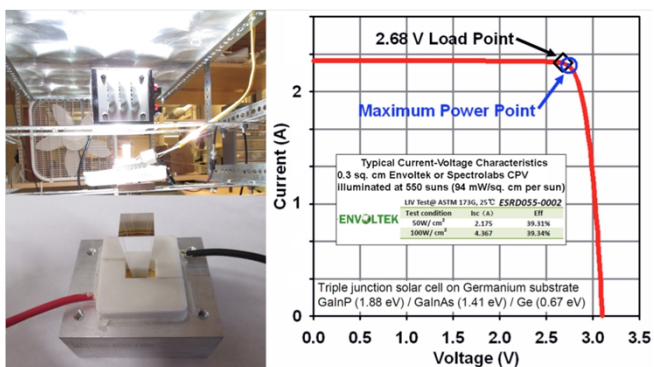
Expanded Figure 3 yield data.

Summarized spectral data.

Outdoor and indoor photocurrent configurations.



Outdoor STEP experimental apparatus. Individual components are detailed in reference 12. The photocurrent is generated by a silicon PV module, and under illumination yields an open circuit voltage: 21.8V; short circuit current (I_{sc}): 0.2A; maximum power voltage (V_{mp}): 18V; maximum power current (I_{mp}): 0.17A; maximum power (P_{max}): 3W; solar efficiency: 18%.



In-lab the photocurrent is provided by a 39% efficient concentrator photovoltaic (CPV) under 1 kW Xenon, daylight color (5600K) AM1(air mass) illumination. Left top: The fresnel concentrator above the AM 1 filter. Left bottom: the unattached CPC under the secondary optical concentrator. Right side: Typical (550 sun) photocurrent - voltage plot of the CPV.

Expanded Figure 3 yield data.

Subsequent to electrolysis, the benzaldehyde yield variation with the electrolysis potential is:

1.02% of 90°C,1V; 1.16% of 90°C,2V; 1.32% of 90°C,3V;
0.14% of 60°C,1V; 0.26% of 60°C,2V; 0.51% of 60°C,3V;
0.09% of 30°C,1V; 0.16% of 30°C,2V; 0.42% of 30°C,3V

Subsequent to electrolysis, the benzyl alcohol yield variation with the electrolysis potential is:

0.25% of 90°C,1V; 0.83% of 90°C,2V; 0.92% of 90°C,3V;
0.1% of 60°C,1V; 0.53% of 60°C,2V; 0.6% of 60°C,3V;
0.02% of 30°C,1V; 0.12% of 30°C,2V; 0.27% of 30°C,3V

Intermediates in Figure 3 are the combined sum of the yield of benzaldehyde and the yield of benzyl alcohol subsequent to electrolysis:

1.27% of 90°C,1V; 1.99% of 90°C,2V; 2.24% of 90°C,3V;
0.24% of 60°C,1V; 0.79% of 60°C,2V; 1.11% of 60°C,3V;
0.11% of 30°C,1V; 0.28% of 30°C,2V; 0.69% of 30°C,3V

Subsequent to electrolysis, the benzoic acid yield variation with the electrolysis potential, as reported in Figure 3, is:

13.7% of 90°C,1V; 25.7% of 90°C,2V; 32.0% of 90°C,3V;
2.6% of 60°C,1V; 8.7% of 60°C,2V; 12.4% of 60°C,3V;
0.5% of 30°C,1V; 1.51% of 30°C,2V; 3.9% of 30°C,3V

Summarized spectral data of the compounds

Benzoic acid (Figure 4, Figure 5):

IR (vmax in cm⁻¹): 1660(C=O stretch), 3200 (O-H stretch), 1400(COO⁻), 2079(-OH, stretching and bending vibration), 1207(C-O stretch), 705(deviational vibration), 667(C-H for benzene ring);

UV: λ_{max} (C₆H₅COOH)/nm 231

Benzaldehyde (Figure 4, Figure 5):

IR (vmax in cm⁻¹): 1660(C=O stretch), 2720 (C-H stretch), 2385(unsymmetrical C-H deformation);

UV: λ_{max} (C₆H₅CHO)/nm 256

Benzyl Alcohol (Figure 4, Figure 5):

IR (vmax in cm⁻¹): 957(C-OH stretch), 2720 (C-H stretch), 2385(unsymmetrical C-H deformation);

UV: λ_{max} (C₆H₅CH₂OH)/nm 213.5

Toluene (Figure 5): UV: λ_{max} (C₆H₅CH₃)/nm 208