Electrochemical Valorization of Carboxylates in Aqueous Solution for the production of Biofuels, Fine Chemicals, and Hydrogen

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1. Supplementary Results

Figure S1. Digital photos of (a) pristine Ti sponge (TS) and (b) Pt@TS.

Figure S2. SEM images of pristine TS at (a) low and (b) high magnifications.
Figure S3. Representative SEM images of *as-prepared* Pt@TS electrodes with the addition of (a) 1 mM, (b) 2 mM, and (c) 5 mM H₂PtCl₆.

Figure S4. Representative SEM images of *as-prepared* Pt@TS electrodes at (a, d) 4 °C, (b, e) 30 °C, and (c, f) 60 °C.
The following reactions took place in the electrochemical cell during the electrolysis:

Anodic reaction: \[2\text{RCOO}^- \rightarrow \text{R-R} + 2\text{CO}_2\uparrow + 2\text{e}^-\] (R1)

Solution reaction: \[\text{CO}_2 + \text{OH}^- \rightarrow \text{HCO}_3^-\] (R2)

Cathodic reaction: \[2\text{H}_2\text{O} + 2\text{e}^- \rightarrow \text{H}_2\uparrow + 2\text{OH}^-\] (R3)

Overall reaction: \[2\text{RCOO}^- + 2\text{H}_2\text{O} \rightarrow \text{R-R} + 2\text{HCO}_3^- + \text{H}_2\uparrow\] (R4)

The electrolyte was acidified with concentrated H$_2$SO$_4$ after electrolysis.

Figure S5. Digital photographs of the solution after Kolbe electrolysis. The unreacted CA and hydrophobic products (i.e. tetradance, heptane, and the isomers of heptene) gathered together on the surface of the solution. An obvious phase boundary was formed between the aqueous solution of bicarbonates and hydrophobic biofuels products.

Figure S6. FE comparison of Pt@TS and Pt/C on TS anodes in the aqueous solution of 0.5 M CA and 0.5 M KOH.
Figure S7. Digital photo of flat Pt electrode after electrolysis test. It could be clearly seen that the surface of the electrode was covered with the organic containment which is consistent with the result from the literature.¹

Reference