

Plasma electrochemistry for organic synthesis: Pinacol coupling as proof-of-concept

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Low-temperature, atmospheric-pressure plasmas in contact with liquids have attracted interest for various chemical applications including the synthesis of colloidal nanoparticles, degradation of organic pollutants, and conversion of abundant feedstocks. Compared to other chemical approaches, plasma-liquid chemistry does not require a catalyst material, is electrified, and produces unique reactive species such as solvated electrons, one of the strongest chemical reducing species.

Here, we present an application of plasma-liquid chemistry to organic synthesis using the example of the pinacol coupling reaction to demonstrate the potential to forge new carbon-carbon bonds. Our study was performed using a direct-current (DC) powered plasma formed between a metal electrode and liquid methanol surface. Parametric studies were focused on methyl-4-formylbenzoate (MFB) as the substrate. The experimental results were supported by a reaction-diffusion model. In addition to the pinacol product, nuclear magnetic resonance (NMR) spectroscopy also indicated methyl 4-(dimethoxymethyl)benzoate, methyl 4-(hydroxymethyl)benzoate, and 4-(methoxycarbonyl)benzoic acid as side products. By varying process conditions such as the initial MFB concentration and the addition of water to the reaction, the faradaic efficiency of the pinacol product is increased up to 80%, and the yield is as high as 46% after 12 h. Scavenger control experiments reveal that the vicinal diol is produced by solvated electron reduction while the side products are formed by reactions with other radicals or the solvent. The generality of the approach is demonstrated by extending to several other aromatic aldehydes and ketones.

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