



Cite this: *J. Mater. Chem. A*, 2016, **4**, 15786

Received 10th June 2016
Accepted 13th August 2016

DOI: 10.1039/C6JA04874a

www.rsc.org/MaterialsA

Highly efficient electro-reduction of CO₂ to formic acid by nano-copper†

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Ultra-fine copper(ii) oxide nanoparticles were used for the electrocatalytic reduction of CO₂ to formic acid at high Faradaic efficiencies. The nanoparticles were directly synthesised via continuous hydrothermal flow synthesis (CHFS) process, which used water as a solvent and reagent. The as-prepared nanoparticles were subsequently formulated into Nafion based inks. For the electroreduction of CO₂, the influence of Nafion fraction on the Faradaic efficiencies and overpotential (for formic acid production), was explored over a wide potential range. The highest Faradaic efficiency for formic acid production (65%) was observed with a 25 wt% Nafion fraction, at a potential of -1.4 V vs. Ag/AgCl. Some insights into the significant increase in Faradaic efficiency for the production of formic acid with the optimum Nafion content, was elucidated with electrochemical impedance spectroscopy.

Introduction

Global warming and climate change have been strongly linked to the increasing concentration of CO₂ in the atmosphere.¹ Over the past 100 years, the amount of CO₂ has risen from 150 ppm to >400 ppm by 2016.² In tandem with CO₂ sequestration and storage, there is a strong drive towards utilising CO₂ to form useful chemicals, as CO₂ is an inexpensive, relatively non-toxic and an abundant carbon feedstock.³ The electrochemical reduction of CO₂ into formic acid, methanol, syngas or hydrocarbons, is an attractive way to utilise captured CO₂ whilst making valuable chemicals.⁴

Copper is a well-known catalyst for converting CO₂ to various products such as methane, formic acid, methanol and CO.^{5–8} However, both polycrystalline copper and single crystal copper electrodes, can also suffer from low activity and require high overpotentials.⁹ In particular, nanostructured copper and its oxides, have been investigated for the electrochemical conversion of CO₂ to chemicals.⁷ The use of oxides, particularly Cu(i), is also of interest as it has been shown to require a relatively low overpotential to convert CO₂ to chemicals.⁸ Typically, nano-Cu (and its oxides) can be synthesised by a range of methods including electrochemical deposition,¹⁰ batch hydrothermal syntheses,^{11,12} precipitation reactions,¹³ microwave assisted¹⁴ and solid state syntheses.¹⁵ Whilst such methods can produce small quantities of catalyst, successful and reproducible scale-up syntheses of nanoparticles are generally more difficult to

achieve in batch processes. In contrast to batch processes, continuous synthesis methods can offer several advantages over batch methods in terms of scalability, ease of processing and allowing independent control over reaction parameters such as reaction time, temperature and pressure. In particular, Continuous Hydrothermal Flow Synthesis (CHFS) using supercritical water is a promising and scalable method for the rapid and controlled synthesis of high surface area metal and metal oxide nanoparticles.¹⁶ A typical CHFS process involves the mixing of superheated water (generally above its critical point $T_c = 374$ °C and $p_c = 22.1$ MPa) in flow with a stream of an aqueous metal salt solution (at ambient temperatures) in a controlled manner. Upon mixing of the two feeds, nanoparticles are instantly formed *via* simultaneous hydrolysis and dehydration under supersaturated conditions. Recently, the use of a confined jet mixer (CJM) was reported by the authors for mixing in CHFS, under highly turbulent conditions.^{17–19} Due to the instant mixing of the reactants and supercritical water within the CJM, nanoparticles can be made consistently and with a narrow size distribution at kg per day rates.^{19–22}

In the electroreduction of CO₂ using nanoparticles, typical electrodes are prepared *via* deposition of the catalyst nanopowder (in the form of a suspended ink) onto a microporous substrate or glassy carbon electrode. When considering the performance of such electrodes, the final microstructure of the resulting catalyst layer can often be overlooked. Recently, proton exchange membrane fuel cell (PEMFC)-like designs were adopted for the electrocatalytic reduction of CO₂, where Nafion ionomer was incorporated in the catalyst layer to help improve CO₂ reduction.²³ Nafion can act as a binder, creating a continuously connected ionic conductive matrix, which allows the reactants and products to be transported between the active catalytic sites and the reactant.²³ Several studies have shown

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† Electronic supplementary information (ESI) available: Details for confined jet mixer, morphology of CuO catalyst layers, analytical characterisation of electrolysis products and impedance spectroscopy model included. See DOI: 10.1039/C6JA04874a

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Table of Contents Reduction Of Copper Oxide By Formic Acid Qucosa

1. Understanding the eBook Reduction Of Copper Oxide By Formic Acid Qucosa
 - The Rise of Digital Reading Reduction Of Copper Oxide By Formic Acid Qucosa
 - Advantages of eBooks Over Traditional Books
2. Identifying Reduction Of Copper Oxide By Formic Acid Qucosa
 - Exploring Different Genres
 - Considering Fiction vs. Non-Fiction
 - Determining Your Reading Goals
3. Choosing the Right eBook Platform
 - Popular eBook Platforms
 - Features to Look for in an Reduction Of Copper Oxide By Formic Acid Qucosa
 - User-Friendly Interface
4. Exploring eBook Recommendations from Reduction Of Copper Oxide By Formic Acid Qucosa
 - Personalized Recommendations
 - Reduction Of Copper Oxide By Formic Acid Qucosa User Reviews and Ratings
 - Reduction Of Copper Oxide By Formic Acid Qucosa and Bestseller Lists
5. Accessing Reduction Of Copper Oxide By Formic Acid Qucosa Free and Paid eBooks
 - Reduction Of Copper Oxide By Formic Acid Qucosa Public Domain eBooks
 - Reduction Of Copper Oxide By Formic Acid Qucosa eBook Subscription Services
 - Reduction Of Copper Oxide By Formic Acid Qucosa Budget-Friendly Options

6. Navigating Reduction Of Copper Oxide By Formic Acid Qucosa eBook Formats
 - ePub, PDF, MOBI, and More
 - Reduction Of Copper Oxide By Formic Acid Qucosa Compatibility with Devices
 - Reduction Of Copper Oxide By Formic Acid Qucosa Enhanced eBook Features
7. Enhancing Your Reading Experience
 - Adjustable Fonts and Text Sizes of Reduction Of Copper Oxide By Formic Acid Qucosa
 - Highlighting and Note-Taking Reduction Of Copper Oxide By Formic Acid Qucosa
 - Interactive Elements Reduction Of Copper Oxide By Formic Acid Qucosa
8. Staying Engaged with Reduction Of Copper Oxide By Formic Acid Qucosa
 - Joining Online Reading Communities
 - Participating in Virtual Book Clubs
 - Following Authors and Publishers Reduction Of Copper Oxide By Formic Acid Qucosa
9. Balancing eBooks and Physical Books Reduction Of Copper Oxide By Formic Acid Qucosa
 - Benefits of a Digital Library
 - Creating a Diverse Reading Collection Reduction Of Copper Oxide By Formic Acid Qucosa
10. Overcoming Reading Challenges
 - Dealing with Digital Eye Strain
 - Minimizing Distractions
 - Managing Screen Time
11. Cultivating a Reading Routine Reduction Of Copper Oxide By Formic Acid Qucosa
 - Setting Reading Goals Reduction Of Copper Oxide By Formic Acid Qucosa
 - Carving Out Dedicated Reading Time
12. Sourcing Reliable Information of Reduction Of Copper Oxide By Formic Acid Qucosa
 - Fact-Checking eBook Content of Reduction Of Copper Oxide By Formic Acid Qucosa
 - Distinguishing Credible Sources
13. Promoting Lifelong Learning
 - Utilizing eBooks for Skill Development
 - Exploring Educational eBooks
14. Embracing eBook Trends
 - Integration of Multimedia Elements

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