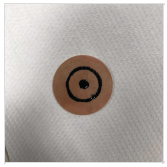

Version 3

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Preparation of ink for electrode deposition via paint brushing using oxide powder V.3

Giulio Cordaro[1]

[1]Université Paris-Saclay, CentraleSupélec, CNRS, Laboratoire SPMS, 91190, Gif-sur-Yvette, France

1 Works for me dx.doi.org/10.17504/protocols.io.btm3nk8n

SOFC Procedures

Giulio Cordaro
Université Paris-Saclay, CentraleSupélec, CNRS, Laboratoire ...

SUBMIT TO PLOS ONE

ABSTRACT

A simple and efficient procedure to produce a viscous ink for brush painting of porous electrodes on top of electrolyte pellets for Solid Oxide Fuel Cells (SOFCs).
The layers have a variable porosity between 20 and 40%, depending on the choice of the calcination parameters, which is strongly influenced on the electrode and electrolyte materials, the particle size distribution and the morphology of the electrode powder.
The thickness of the layers is variable in the 10-100 µm range, depending upon the amount of ink deposited.
All the calcination parameters, the electrode and electrolyte materials, the particle size distribution and the morphology influence the adhesion of the electrode layer on the electrolyte substrate.

ATTACHMENTS

Know-How_P01_preparation-of-ink.pdf

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KEYWORDS

Electrode, Paint brushing, Viscous paste, Ink, SOFC, Solid Oxide, Terpineol

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The calcination parameters should be optimized for each sample, to ensure a good adhesion of the layer, while meanwhile to avoid the production of a insulator phase at the interface due to a parasitic reaction between electrolyte and electrode. In addition, the electrochemical performance of each sample can be strongly influenced by the calcination.
First, the reactivity between electrode and electrolyte can be evaluated with a common reactivity test: prepare a 50:50wt. mixture of the powders, calcine in a furnace at different temperature and analyse with XRD to ensure that no additional peaks are present in the spectra after the calcination.
Once the ink is painted and calcined, the adhesion can be quickly evaluated with a small piece of tape: if the layer is removed with tape, the adhesion is poor, hence it is not worth to perform the electrochemical measurements.
The optimal calcination parameters are identified as those producing the best electrochemical performances among a series of different samples, being careful to avoid as many changes in the preparation as possible.

1. Preparation of organic viscous paste 3h

1 Weigh terpineol directly inside the bottle **1.52 g or rescale all ingredients if need different amount**

Glass bottle with plastic lid and magnetic stir bar inside for paste preparation

A	B	C	D
Compounds	**CAS Number**	**Percentage [wt./wt.]**	**Amount [g]**
Ethyl-cellulose	9004-57-3	4%	0.0800
Terpineol	95-55-5	76%	1.5200
Iso-propanol	67-63-0	20%	0.4000

Massive ratio of compounds for the paste production

1.1 Add iso-propanol **0.4 g**

1.2 Close the lid and stir few minutes on a magnetic plate

1.3 Weigh ethyl-cellulose separately on tin foil **0.08 g**

1.4 Add the ethyl-cellulose little by little to avoid big agglomerations

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Be careful to limit the solid on the walls and on the top of the stirrer

1.5 Close the lid and stir until complete dissolution and homogenization
Be careful that there are no clumps on the walls

2. Treatment of the electrode powder for ink preparation 6h

2 The electrode powder ideally will be treated previously to ink preparation to avoid the presence of big agglomerates

2.1 Ball milling at 300 rpm for 4 hours with balls/powder mass ratio of 10 to 20 with ethanol as media (covering powder), with zirconia or WC jar/balls
Be careful that milling hard materials can pollute the powder with worn particles from balls or jar

2.2 Rinse with ethanol and recover the powder in a beaker, then dry on a hotplate or in a muffle at 80°C

3. Mixing of organic vehicle with powder (powder:paste = 60:40 wt.) and painting 30m

3 Clean and weigh a plastic bottle with cone-shaped bottom

Plastic bottle used to prepare inks

4 Weigh about 0.2 g of viscous paste with a thin metal tip (Figure Step 1)

5 Calculate the amount of electrode powder to add (ratio 3:2 in weight)

6 Weigh, add the electrode powder and mix with the metal tip to perfectly homogenize the ink
Be careful to completely incorporate all the powder

7 Rest for sedimentation at least 15 minutes

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8 Dip the brush only in the top part of the ink and paint the electrolyte surface as more homogeneous as possible

9 Dry at 150 °C for 3 hours and paint the other side

10 Calcine at 1000 °C for 2 hours (±1 °C/min) and check the adhesion!
Be careful that these values are indicative. The optimal parameters need to be evaluated for each sample, depending on the choice of electrode and electrolyte materials, and on the particle size distribution and the morphology of the electrode powder.

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