Influence of sonication on co-precipitation synthesis of copper oxide catalyst for CO_2 electroreduction

Daniela Roldán[1] , Hilmar Guzmán[1,2] , Nunzio Russo[1] , Simelys Hernández[1,2]

1. Applied Science and Technology (DISAT), Politecnico di Torino, Torino, Italy.

2. Istituto Italiano di Tecnologia, Center for Sustainable Future Technologies, Torino, Italy.

CONTROL ID: 3501687
daniela.roldan@polito.it

Politecnico di Torino
1859

CREST
Catalytic Reaction Engineering for Sustainable Technologies

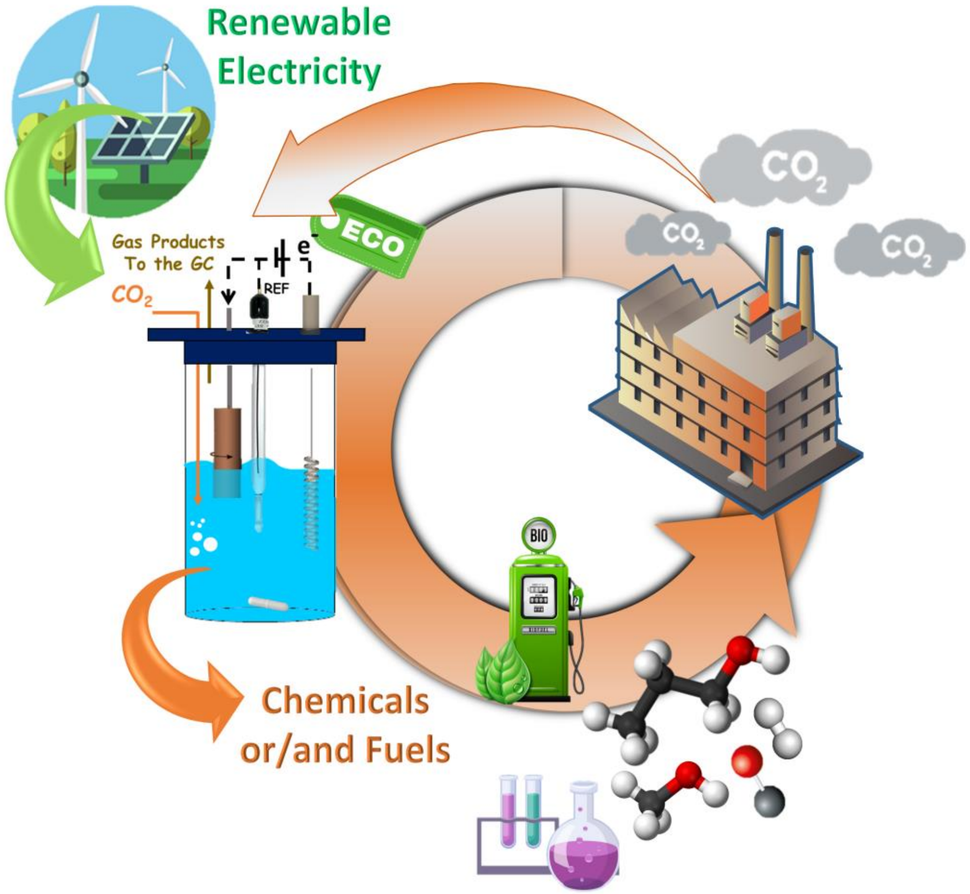
2021 VIRTUAL MRS®
SPRING MEETING & EXHIBIT
April 18–23, 2021

eni

Overview

The need to reduce greenhouse gas emissions and increase our energy supply makes the **electrochemical reduction of CO_2** (CO_2R) a very attractive alternative to produce non-fossil-based fuels or chemicals. **Copper-based catalysts** is one of the catalyst that most efficiently promote the formation of species with one or more carbon-carbon bonds from the electrochemical reduction of CO_2.

It was decided to evaluate the **effect of the ultrasound application (US)** on the shape and size of the particles obtained, its electrocatalytic activity and its selectivity to products of interest.



Physical characterization was carried out by using different techniques including **X-ray diffraction**, **BET** and filed-emission scanning electron microscopy (**FESEM**).

Electrochemical tests for CO_2 reduction were done under ambient conditions.

Materials and Methods

The catalysts were synthesized by precipitation method with different ultrasound conditions to evaluate its effect on the performance of the catalysts. All catalysts are copper-based and with the **same precursor concentration**. **Different ultrasound amplitude** values were applied:

Table 1. Conditions of the different catalysts.

Catalyst name	Precursor concentration, M $Cu(NO_3)_2.3H_2O$	Ultrasound conditions	%Amplitude
Cu-06	0.6	Without ultrasound	-
Cu-06-%23-A	0.6	Ultrasound in the ageing process	23
Cu-06-%30-A	0.6	Ultrasound in the ageing process	30
Cu-06-%37-A	0.6	Ultrasound in the ageing process	37

4 catalysts with different US conditions

The catalysts were synthesized by **precipitation method** :

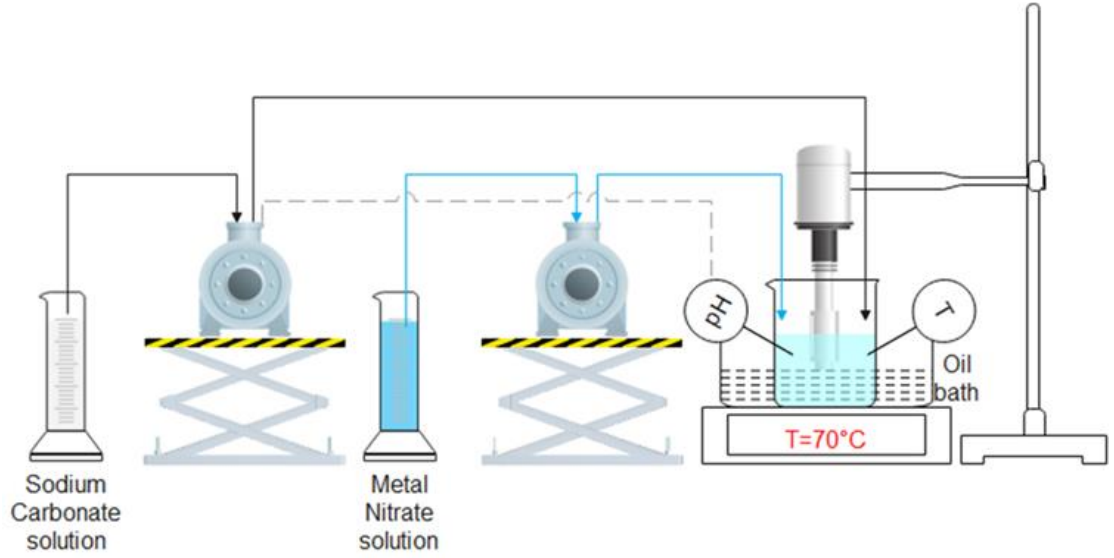
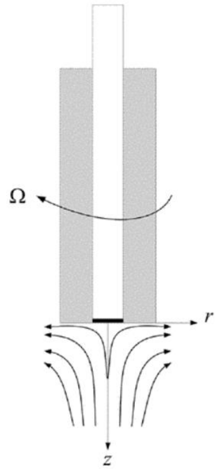


Figure 1. Synthesis process set up.

The tests were carried out in a three-electrode electrochemical cell using a **rotating disk electrode (RDE)** system.



Results

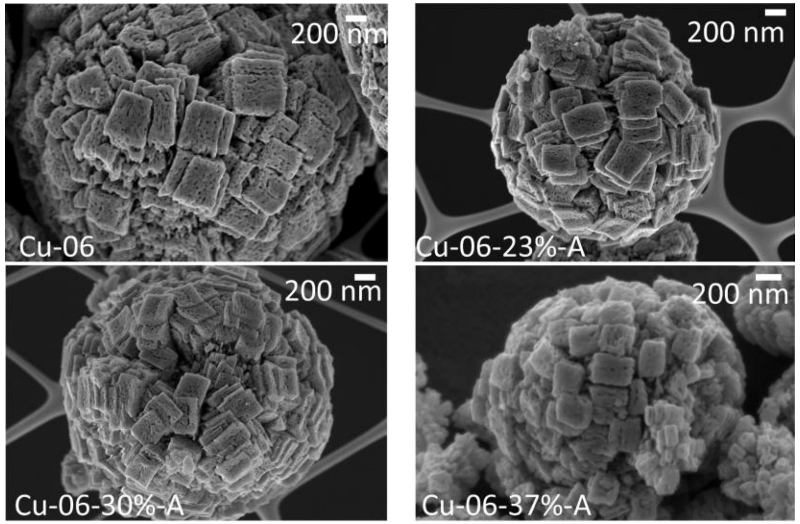


Figure 2. FESEM images of Cu-based catalysts.

The US during the ageing process do not influence the final morphology of the catalyst at the different applied amplitudes. The highest US amplitude produced less spherical particles.

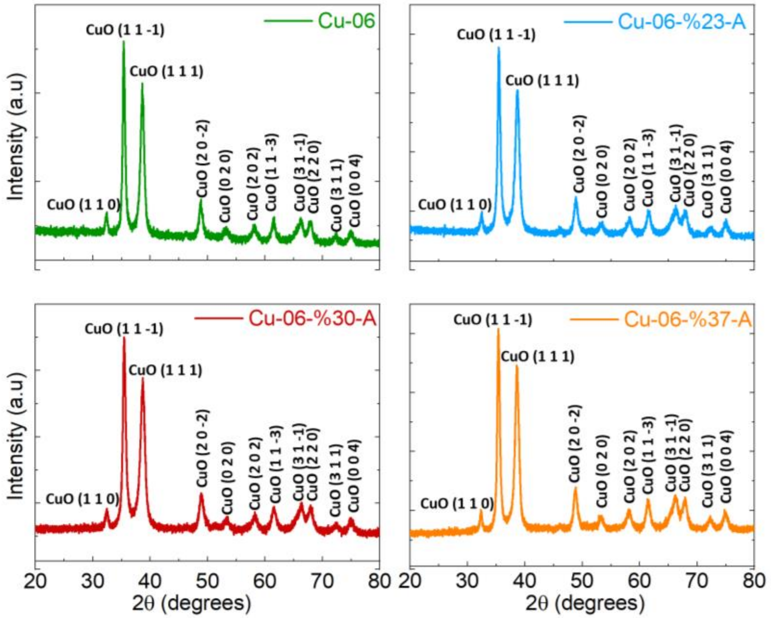


Figure 3. XRD of Cu-based catalysts.

The crystalline structure is attributed to **CuO** and does not change with the US irradiation.

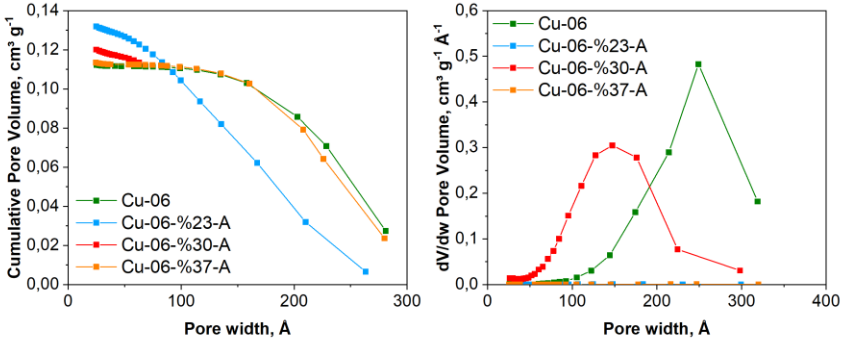


Figure 4. Pore size distribution.

Pore size distribution is narrower by assisting the co-precipitation synthesis with US

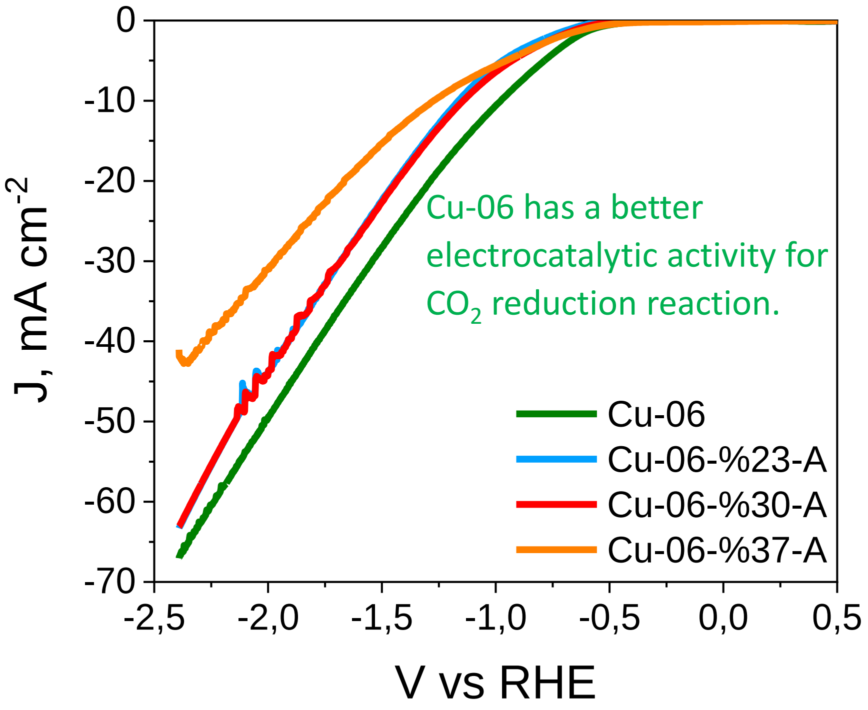
Table 2. Physical-chemical properties.

Catalyst	BET surface area, $m^2 g^{-1}$	Total pore volume, $cm^3 g^{-1}$	Crystallite size, nm (1 1 -1) facet of CuO
Cu-06	18.40	0.107	13.97
Cu-06-%23-A	34.07	0.135	11.38
Cu-06-%30-A	32.07	0.125	11.39
Cu-06-%37-A	18.68	0.124	16.78

The US has **increased the surface area** of the sonicated sample at %23 and 30% in comparison to the non-sonicated one. Probably the smaller BET surface area of the %37 sample is due to the higher crystallites size. On the other hand, there is an increase of pores volume by using US compared to the original sample, Cu-06.

Results

Cu-06-23%-A and Cu-06-30%-A exhibit the same performance, indicating that the prepared materials have similar electrocatalytic activity for the CO_2 reduction reaction.



Cu-06 has a better electrocatalytic activity for CO_2 reduction reaction.

Figure 5. Linear sweep voltammetry (LSV).

The most promising catalyst among the new US-prepared catalysts is the **Cu-06-%30-A** with **14% of FE to alcohols.**

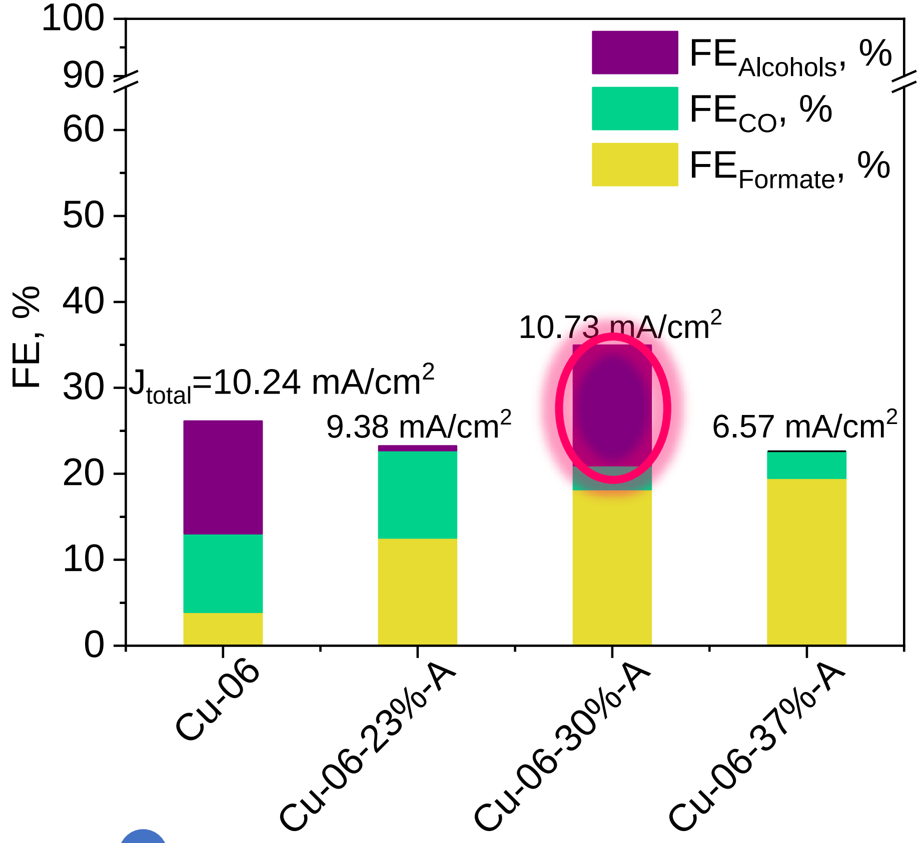


Figure 6. FE towards the main high-value products.

The use of ultrasound **favoured the selectivity towards formate.**

Conclusion

Cu-based catalysts with different physicochemical properties were prepared by ultrasound assisted co-precipitation method.

- Regarding the physical characteristics, we found that **pore size distribution is narrower by increasing the US amplitude**. On the other hand, there is no significant difference in morphology and dimension of particles.

- **The surface area increased with the use of ultrasound** (23 and 30%), which is attributed to an improved dispersion created by acoustic cavitation. However, **there is an optimal amplitude (30%)** over which the advantages of the use of ultrasound are diminished.

- Ultrasound has also an effect on **Copper-based catalysts performance**; in this case, the selectivity towards alcohols and C_1 products (Formate) was enhanced.

This work has been performed with the financial support of Eni SpA and the R&D Program Energy Transition (Cattura e Utilizzo CO_2).