

Improvement and utilization of a HPLC system for studies of electro-oxidation of Glycerol.

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Abstract

The oxidation of glycerol to value-added products is very important from the economic point of view. In this work we performed HPLC experiments with the most important products of the oxidation of glycerol containing three atoms of C.

Key words: HPLC, Glycerol, Electrocatalisys.

Introduction

This Project coincides with the establishment of a new research group in the State University of Campinas, Institute of Chemistry in the area of Electrochemistry, more specifically Electrocatalysis. This group is devoted to study the electrooxidation of organic molecules derived from Biomass resources to produced value-added products. The most important part of the project is connected to the separation and quantification of the product of the electrooxidation of glycerol (GIOH). It can, in principle, be done by using HPLC which is currently successfully used by the groups of electrochemistry of the University of Poitiers (France)¹ and Leiden University (The Netherlands)².

We are setting the conditions of our HPLC systems for the separation of the products of the oxidation of GIOH. The second step will be to generate calibrations curves to turn the method quantitative. Finally, the set up will be used to separate and quantified products of the oxidation of GIOH generated through electrolysis using different experimental conditions over carbon supported metallic nanoparticles with different composition.

The electrooxidation of GIOH (and other molecules) are being studied in conventional electrochemical cells by cyclic voltammetry and FTIR in situ

Results and Discussion

The HPLC experiments are being carried out in an Agilent Technologies 1200 Series HPLC equipment and the column Bio-Rad Aminex® HPX-87H 300x7.8mm.

Our starting point is to use standard conditions for this system, i.e., H₂SO₄ 5mM as eluent and 50°C (maximum temperature for our equipment). Besides, we starts using the most important GIOH oxidation product containing 3 carbon atoms, i.e., DHA (Dihydroxyacetone), Gld (Glyceraldehyde) and the GA (Glyceric Acid).

Image 1 shows the chromatograms obtained by injecting 50 microliters of solutions 1mM of: the eluent (blank), GIOH, GA, Gld, DHA and a mixture of all of them. In every chromatogram we can advertise the injection peak at around 6.9 min and the corresponding peak generated for each compound. We observe very well-developed peaks, except for the case of GIOH. The chromatogram for the mixture is the sum of the features presented for all of the others chromatograms, except for the case of GIOH.

The chromatogram of the mixture shows that we are able of identify the presence of each of the product in

the mixture, however we cannot solve the peaks of Gld and GA.

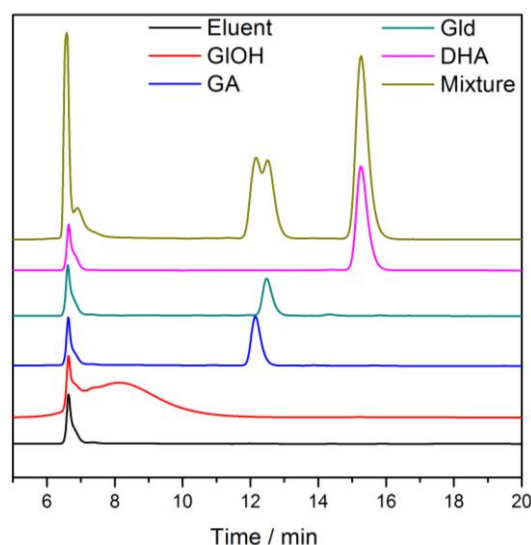


Image 1. Chromatogram of the standard products of GIOH.

Conclusions

We succeeded in our first attempt to set the parameters of our HPLC system. We are now repeating the experiments with GIOH and obtaining calibration curves for all of the substances.

The next step will be to repeat the experiments but changing the concentration of H₂SO₄ of the eluent in order to improve the separation of GA and Gld.

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¹ Holade, Y.; Servat, K.; Napporn, T.W.; Kokoh, K.B. *Electrochimica Acta* 162 (2015) 205–214

² Kwon, Y.; Hersbach, T.J.P.; Koper, M. T. M. *Top Catal.* 57, 14, 1272-1276, (2014).