Continuous Process for the Oxidation of Alcohols using Electrochemically Synthesized Persulfates

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Introduction

With increasing pressure to improve the cost effectiveness and sustainability of chemical processes, there is a need to develop continuous synthetic processes. Organic reactions carried out using water are of great current interest in this respect. Persulfates are a source of readily accessible oxidizing agents. They are commonly used in industry for bleaching [2] and for wastewater treatment [3]. However, examples of their application in chemical synthesis are limited. Oxidation of alcohols to carbonyl compounds is an important reaction that is particularly difficult to scale up.

We will describe a novel approach to this problem, by using electrochemistry to generate oxidants (persulfates) for a continuous flow process.

Experimental

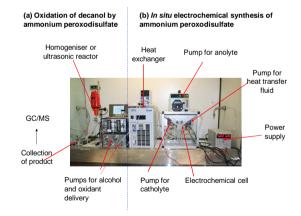


Figure 1. Experimental configuration for oxidation of alcohols using a multiphase reactor via electrochemically synthesised inorganic peroxo compounds

The oxidation of decanol or decanal in toluene by electrochemically-synthesised ammonium peroxodisulfate (aqueous solution) was realised in a continuous flow system depicted in Figure

1. For the electrochemical synthesis of ammonium peroxodisulfate, a continuous twocompartment electrochemical cell was used consisting of a boron doped diamond anode, a nickel cathode and a semi permeable membrane (NafionTM424). Equimolar ammonium sulfate and sulfuric acid solutions were employed for both the anolyte and the catholyte. The temperature of the electrochemical reactor was controlled at 10°C to suppress undesirable reactions at the anode. A 0.5M ammonium peroxodisulfate solution was continuously produced at 0.5 ml/min and a current density of 0.15 A/cm². This solution was contacted with a solution of 0.2M decanol in toluene using an ultrasonic reactor, to facilitate the generation of large interfacial areas via emulsion formation.

Results/Discussion

Figure 2 shows the conversion of decanol to decanal and decanoic acid as function of reactor temperature. The selectivity between the two oxidised products is also dependent upon temperature, details of which will be discussed later.

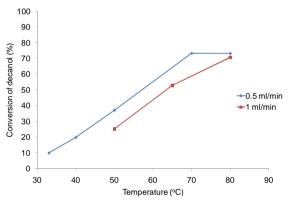


Figure 2. Effect of temperature on the conversion of decanol to decanal/decanoic acid, using ammonium persulfate as oxidant.

References

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