A STUDY OF ORGANIC MATERIALS ON ELECTRO SYNTHESIS

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Abstract

Despite its long history, electrosynthesis has never been a common practice for the production of organic compounds in organic synthesis laboratories or in industry. One of the main reasons for this is the nature of the references. This review highlights the demand of electrosynthesis users and explains it for articles. It is attractive for synthesis chemists to demonstrate the high conversion of the reactant to the product, as well as the separation of the pure product at the desired scale and high efficiency. In addition, especially the electrolysis cell (geometry, dimensions, components and resources, mass transfer regime, etc.) and all control parameters (solvent, reagent concentration) and Electrolyte, pH, cell flow, temperature, etc.) were mentioned.

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1. Introduction

Electrosynthesis of organic materials has a history of nearly 200 years and there are many references in this field. Over the years, many books and reviews (recent examples) have consistently covered various electrodes in chemistry, but electrolysis is still a method used in the laboratory and industry to prepare organic matter compounds. Why is that? One reason is its nature in references. Electrosynthesis of organic matter is poorly provided to organic chemists. Electrolysis of organic compounds needs to be recognized for several separate purposes.

• Help interpret voltammetry

• Suggest a route for in vitro synthesis of an organic molecule.

• Build a trade route to create an organic compound

The tests required for each are significantly different, and many authors do not clearly state the purpose and reasons for the electrolysis reports in their articles. For example, there is a big difference between demonstrating an electrode reaction and electrolysis proving to be a useful in vitro synthesis. Worst of all, books and reviews often fail to differentiate, making these references misleading and confusing to non-electrochemists. Additionally, attempts to replicate an electrosynthesis in different laboratories often lead to low selectivity and / or efficiency. Most of these situations are due to insufficient details in the electrolysis cell (geometry, dimensions, material composition, mass transfer distribution, etc.) and electrolysis conditions (solvent, reactant and electrolyte concentration, PH, temperature flow cell, etc.). Of course, it is very important to follow the information in this article exactly. Therefore, in this study, experimental results were defined for different purposes and repairs that led to the introduction of a widely used synthetic method were proposed.

2. Interpretation of Voltammetry

Voltammetry of organic molecules can be performed to develop an analysis method, to understand the effect of solution or electrode materials on the rate or selectivity of an electrode reaction, or to describe the mechanism and / or kinetics of a homogeneous chemical reaction. It is clear that electrolysis is useful under similar voltammetric conditions. Therefore, electrolysis can often use a low concentration of reactants causing a low cell flow, and a high electrolyte concentration makes it possible to use a three-electrode cell and control static and electrode potentials and

electrode potentials. As with voltammetric experiments, it also makes sense to limit the electrolysis time only to low reactant consumption and minimal changes in the solution medium.

3.Laboratory Synthesis

In this lab, an electrolysis will compete with other methods to convert the raw materials into the desired product, and actually synthesize the desired product with other raw materials. It should be emphasized that just showing the conversion of some reactants to a product is not a "synthesis"; Synthesis is the method of producing the desired amount of pure product. Organic chemists are aware of the amount they need (usually 10 mg to 100 g) and adopt this approach with increasing emphasis due to the availability of raw materials, easy synthesis conditions (including availability of suitable equipment) and selectivity. There is no doubt that electrolysis can be an environmentally friendly method. This method allows oxidation / reduction without the use of stoichiometric amounts of redox reactants and / or toxic or hazardous reactants, and the overall conversion is done close to ambient temperature and pressure. Synthetic chemists expect good selectivity up to 1.0 relative yield as well as high conversion of the reactant to product. Moreover, the simple separation of the pure product increases the attractiveness of this method from the synthesis requirements, there is a significant reduction in the reactant concentration associated with the production of the product.

In most electrolysis, the conversion is accompanied by other major changes in the electrolysis environment and is often accompanied by the accumulation of protons or hydroxide. For a successful synthesis, the conversion should not be affected by changes in the solution composition, or the synthesis should include steps to minimize the changes. It is important to know that even with a relative flow efficiency of 1.0, formation of the required amount of product for correct synthesis

produces significant cell flow (eg 2e - reactive molecule containing the reaction electrode). The use of such cell currents probably requires cells with substantial size electrodes. The dimensions of the electrode depend on the current density and a current density of <50 mA cm-2 should be considered to reduce the size of the equipment. Since the current density depends on the concentration of the reactant and the mass transfer regime, an acceptable solubility of the reactant (<0.1 M) and mixing or efficient flow in solution are required. Cell design requires electrode geometry and potential distribution on the working electrode surface, the effect of the chemical composition of the counter electrode and the mass transfer regime. Such cellular currents go beyond commercial pansiostatic statistical capability. It does not matter, however, because laboratory currents and potentials, which are not independent parameters, and laboratory electrolysis are easily controlled by the available low-cost power supply, but it can be beneficial to reduce cell flow during electrolysis. In a laboratory synthesis, only reaction selectivity and high product efficiency are essential. It is therefore vital that electrolysis is carried out in a situation where the electrode reaction leads to cleaning of the reaction interface and this interface is disrupted by a dominant pathway to the product. The reactive nature of electrode materials, solution composition and temperature can affect the selectivity of the reaction. It is important to acknowledge that organic matter synthesis likely involves multifactorial substrates, and therefore we need to be aware of other functional groups in the reacting molecule. Currently, simple compounds with a single active group have been extensively studied. Water is an ideal solvent for electrolysis. Electrolytes are highly soluble and these aqueous solutions are highly conductive. In addition, the electrochemical oxidation / reduction of water (which can occur as a competitive reaction at the working electrode or as the main reaction at opposite electrodes) leads to gas formation and pH change. Organic solvents are more attractive to synthesis (higher solubility of organic matter, etc.). Unfortunately, the conductivity of electrolyte solutions in organic solvents varies significantly, but rarely depends on organic solutions. This

increases the cell voltage (not necessarily a problem), but results in a greater potential distribution across the working electrode surface; For example, the subsequent use of 3D electrodes has been limited to increase cell flow. In addition, electrode reactions in organic solvents are often complex, and aprotic solvents especially exposed to acid / catalyst catalysts decompose homogeneously. These factors can lead to a contaminated product solution that prevents the pure product from decomposing. The use of aqueous / organic mixtures or alcohol or carboxylic acid as solvents for oxidation can lead to increased compatibility. Dissolved anodes include syntheses that can help reduce the cathode in APKROTIC solvents. Platinum is commonly used for anodic oxidations in references, but these electrodes are very expensive in the dimensions required for synthesis. Graphite or carbon polymer composite is a more rational choice. A wider range of materials are available for cathodic reduction. For example carbon, lead, steel, nickel and copper. Most of these materials are available in plates as well as meshes and foams with many possibilities for cell design. Ease of synthesis means that the necessary equipment (cells, electrodes, control electronics) is easily accessible. Human cells immediately take the lead. All laboratories have different volumes of people, and it is interesting to assume that electrolysis requires only two electrodes to be immersed in a human, and perhaps a coating to maintain the oxygen environment. Unfortunately, human cells do not work well at all. Achieving a high ratio of the surface area of the active electrode to the volume of the solution and efficient mass transfer, high selectivity and conversion, as well as high cell flow is possible during logical synthesis. The completion of the electrolysis in minutes is attractive for a maximum of a few hours. In addition, the electrolysis time can lead to a competitive homogeneous chemical reaction (especially hydrolysis or sololysis) with the electrode.

Another approach is to make cells based on microscopic plates, but these cells are only suitable for a traditional few milligrams. This is the number of output

cells (eg Electro cell family, Electrosyl cell, Flow-C cell) that can be used with replacement of cells, purchase of a commercial cell and reagent solution recovery. It passes the reactant solution through one route, giving the product only through an enlarged electrolysis channel that allows high reactor conversion. (Eg Amriite family Syrris cells) However, it is important to recognize that each electrolysis cell is only designed to produce the product within a certain range of values; For example, ammonite cells are designed to be synthesized in an amount of 0.1 to 50 grams per hour. A significant increase in scale may require a change in cell design. Therefore, before purchasing a cell, it is important to consider the amount required to produce the product and the importance of high conversion of the reactant to the product. Of course, the operation of commercial flow cells can be replicated using laboratory equipment, and the production of electrolysis cells takes effort and time. A number of cell designs with different geometries are described. Ease of synthesis is often defined by direct separation of the product. This process results in complete conversion of the reactant to the product and the absence of high concentrations of electrolyte in the reaction medium. Such efficiency helps to use cells with long channels and cells or narrow gaps between electrodes. Flow (charge) efficiency is important in laboratory synthesis only when competitive reactions (oxidation / reduction of reactant to alternative products or solvent / electrolyte) lead to loss of selectivity or complication of pure product decomposition; Energy efficiency is never about lab scale. A proper synthesis also requires chemical examination of another electrode. The term charge is the same amount of chemical change on the counter electrode as the working electrode and it is important that the counter electrode's chemistry does not degrade the performance of the working electrode. In addition, the F2 charge path in the working electrode causes significant changes in the composition of the electrolysis solution, typically acid or base formation (usually greater than M1) at a cathode. A common strategy uses a split cell. The chemical composition of the opposite electrodes provides a constant PH in the electrolysis solution that can promote competitive chemistry. Other strategies for

cross-electrodes in an uninterpreted cell include 'pair synthesis' and 'dissolving metal anodes'. The anode and cathode chemical compounds can be separated alternately with an ion-permeable membrane or a porous separator. However, it should be noted that the ion transfer between the anode and cathode is a prerequisite for electrolysis in the cell, and when there is a concentration gradient some transfer of reactant and product occurs between chambers. In addition, suitable spacers can be a source of trouble and in practice the separator / membrane is always defective and can complicate cell design. An absolute requirement for an article is to suggest laboratory synthesis with detailed descriptions of the method, particularly electrolysis equipment. This should include a complete description of the cell design, cell dimensions, electrodes (raw materials, pretreatment, geometry, position and dimensions), separator if used, solution volumes, concentration of both reactants, and product as well as electrolyte, temperature, cell transfer regime and cell content. should isolate and define. Defining the mass transfer regime in a mixed solution requires expressing the rotation speed and the position and shape of the mixer. When in a flow cell, flow velocity, channel dimensions, inlet and outlet solution design, and presence / design of turbulence enhancers should be specified. Without such accurate information, synthesis cannot be successfully transferred from one laboratory to another.

4. Commercial Production

When using electrolysis at a scale of> 1,000,000 tonnes per year for the hydroderification of acrylonitrile to adiponitrile. The organic compound market is generally very small. . The required scale of production in the pharmaceutical, agricultural, food and chemical industries increases from 1 to 100 kg per day. This requires cell currents in the range of 20-5000 amperes and electrode areas in the range of 200 cm2-50 m2. The parallel plate cell design is likely an available electrolyser, but other designs such as the BASF bipolar disc cell have been used

commercially. With a pair of electrodes, a lower scale can only be achieved in a virtual plate flow cell, while a higher scale can be achieved for such a set of cells in a stack. There are several driving forces for the development of electro-organic industrial processes. This includes (a) lowering the cost of the product, (b) reducing the number of steps in a multi-step synthesis, and (c) reducing the risks associated with its production. This requires replacing toxic / hazardous reactants, including many organic solvents, avoiding diffusion streams of spent reactants, and operating at ambient temperature / pressure. Another possible consequence for the reactant of an electrolytic process is the reduction in the amount of by-products introduced. Recovery of solvents and / or electrolytes is an advantage. Obviously, an imperative for the development of a new industrial process is the possibility of its usefulness. This product must have a stable or developing market and can be sold at a much higher price than reactant. Reactors should also be available. High selectivity and conversion (although not necessary after the reaction solution has passed through the cell) as well as an easy method of separating the pure product are important. In fact, strongly affected electrolysis conditions require that product separation requirements be met. For example, an electrolyte with specific properties (eg, easily extractable or distilled) can be chosen. Once the conversion of the reactant to the required product is complete, the processes can run in batches where the reservoir solution solution is continuously removed to separate the product. Complete conversion can be accomplished using a three-dimensional electrode with a long channel cell or a recycling system as an interruption in reactor omission, preferably increasing the electrolysis time with a planned reduction in cell flow. . Experience (or calculation of Farabi's law) shows that when process economy is very important, electricity consumption will be very low, most likely the investment cost for electrolysis cells will prevail. Since the size / number of cells is determined by the current density, the target center should be achieved when the current density is ideally close to 100 mA cm-2. It is also recommended to avoid expensive electrode materials and spacers. Platinum is a start and carbon, lead and steel electrodes are

also attractive. Undivided cells not only avoid the blocker but also reduce the number of cellular components and assistive devices (such as pumps and tubes). It is essential that this technology works consistently over a long period (months, respectively) without changing the electrodes and separators. Electrodes are subject to corrosion and wear and their stability has always been taken into account. While the development of a business process is likely to follow a successful lab synthesis, a successful lab synthesis may not always be scaled up and a variety of challenges may be met. Therefore, laboratory synthesis and industrial electrolysis should be considered as related but separate activities. Inevitably, cell technology will be different. In developing industrial processes, the selection of cells available on the open market is beneficial and their scale depends on the demand of the product. Again, parallel screened cell designs easily meet these demands. Experimental procedures should be performed in the selected cell as soon as possible. It is an opportunity to design a new process, transform the reactant into a product and re-examine the electrolyte selection of the electrodes, as well as design a completely new package to try to combine single processes. The form (pure or suitable solution for more chemical composition) of the reactant present and desired for the product is known as its characteristics. Stability of performance is important (and the stability of electrodes and other cellular components is an issue to be considered) and the combination of unit operations would be highly desirable. When choosing reactions for development, (a) products with stable and possibly broad markets (b) conversion with significant increases in quantity and (c) recently produced products by a technology with significant barriers; For example, based on toxic / hazardous agents or generating significant streams from by-product. In the past, targets were chosen mainly on the basis of difficulties in finding redox reactants and conditions suitable for highly selective conversion. If reaction with chemical reactants is impossible, it is unlikely to occur at very selective electrodes.

5. Conclusion and Discussion

Electrochemical oxidation and reduction of organic molecules have been reported. Only a few articles can claim to report a "synthesis". Usually the authors tend to show the formation of products (usually still in the electrolysis solution) without having to act to separate the pure product, which is a useful amount. "Synthesis" is the request of other chemists to prepare a product by a known method that requires detailed descriptions of all steps. When this method involves electrolysis. A detailed description of the cell used is important; Cell design, geometry, and dimensions make the vaccine performance at least as effective as electrode materials and volatile soluble conditions, but are widely overlooked for systematic study in references. It is not sufficient to describe the cell type and electrode material; Complete description of the electrolysis cell and its operating conditions, including cell type, cell dimensions, electrodes (materials, pretreatment, geometry and dimensions), solution volumes, both reactants and electrolyte concentration, temperature and cell flow, and all parameters that determine the mass transfer regime (Rotational velocity and flow velocity and mixer position, cell inlet and outlet design, presence of turbulence actuators) should be considered. Articles should also consider methods that are more useful than competing methods. Unfortunately, "synthesis" would only be suitable to create a certain amount of product. Increasing the amount of product with increasing cell size, reactor concentration or electrolysis time has only a limited effect. A significant increase in product quantity requires redesign of the electrolysis cell and operating conditions. It would be very useful if all articles reporting on the electrolysis of organic compounds have a clear view of the purposes of electrolysis. If the purpose of a concrete "synthesis" is to prepare a significant amount of product, it should be disclosed, including a complete and accurate description, including cellular conditions and electrolysis. . The future of electrosynthesis will be developed directly through the publication of more essential articles and the interest of organic

chemists and critical evaluation of existing references. The author of the book / review should be very clear about which articles actually report a synthesis.