

Electrocatalytic Hydrogenation Reaction of Chalcone

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Introduction The chalconoids constitute an important class of naturally occurring flavonoid compounds, exhibiting a wide spectrum of biological activities, which include potential applications as artificial sweeteners, new drugs or agrochemicals [1]. The most common and widespread compounds of the chalconoid group are the chalcones, which possess a 1,3-diaryl-2-propen-1-one carbon framework [2]. However, there are also other important derivatives of this family, such as the dihydrochalcones [3]. Our laboratory began with the naringin extraction and its possible use for preparing the corresponding dihydrochalcone. The naringin-chalcone equilibrium in an alkaline media was studied and preliminary results of the chemical and electrochemical hydrogenation were obtained [4]. The electrocatalytic reaction can be carried out in more moderate conditions of pressure and temperature compared with the classic catalytic one. Up to present the efficiency of the hydrogenation reaction do not overcome 38%. With the objective of optimizing the conditions of the electrocatalytic hydrogenation of naringin chalcone, other chalcones are synthesized with different substitutes in unsaturated carbon-carbon bond. In relation to the stability of chalcones in an alkaline media, it is known that the cleavage was observed with sodium hydroxide concentrations higher than 0.3M leading to a mixture of benzaldehyde and acetophenone as products [5]. Taking into account the decomposition of the chalcone in an alkaline aqueous media this work study the electrochemical reduction of chalcone in an ethanolic media on palladium and palladinized palladium electrodes. Experimental The chalcone was synthesized from acetophenone and benzaldehyde [6] in ethanol-water potassium hydroxide solution. The electrocatalytic hydrogenation was performed using a cell with two compartments [7, 8]. A palladium sheet (thickness 50 μm) served as a working electrode for water electrolysis to produce H atoms and as separator between the electrochemical and chemical reaction compartments. One compartment was used for the electrochemical production of H atoms in a galvanostatic electrolysis of water in a 6 M KOH solution. The counter electrode was a Pt sheet. On the other compartment with palladium chloride in HCl solution for the palladinization process or chalcone solutions for hydrogenation reaction was used. The palladinization was carried out chemically using active hydrogen, which was produced in a galvanostatic electrolysis at 25 mA for 1h followed by passing through the Pd sheet electrode. The palladinized Pd sheet electrode was washed with distilled water and after that it was used for a hydrogenation of chalcone. For the hydrogenation of chalcone the compartment contains a 10⁻⁴ M chalcone in alcoholic media. The hydrogenation product was analysed at different reaction times us-

ing UV visible spectra and chromatographic analysis. Results and discussion. The structure of the chalcone skeleton corresponds to a compound synthesized from acetophenone and benzaldehyde. Usually the molecule has an A ring (with carbonil group) and a B ring. The UV spectra of chalcones are characterized by an intense band I and a diminished band II absorption [9]. The major absorption band in chalcones (band I) usually occurs in the range 340-380 nm and the band II is usually a minor peak in the range 220-270 nm. The UV spectra of chalcone that we synthesized shows an intense band at 310 nm and two diminished bands at 270 nm and 230 nm. As the reaction time increases, the band at 310 nm decreases and a band at 240 nm appears and increases. The main chromophorous groups of the chalcone structure, which are responsible for the UV absorption, principally correspond to the carbonil group in A ring and to the double carbon-carbon bond. With hydrogenation of the double bond the main active group is the carbonil united to A ring, responsible of the band at 240 nm. The spectrum of the acetophenone was also measured and it shows its main band at 242 nm which gets superposed with the spectrum of the hydrogenized chalcone. Therefore the hydrogenation reaction was followed at first by measuring the visible UV spectra at different reaction times. The diminution of the chalcone absorption vs times gives us a measure of the hydrogenation reaction time. Absorption vs time experiment was performed using the palladium sheet and the palladinized palladium sheet as a catalyst. The increases of the catalyst surface is beneficent for the hydrogenation reaction. Taking into account the absorption values, after 43 hs, the yield is approximately 87% are studied using chromatographic and RMN analyses. The study of the hydrogenation reaction is performed with the other chalcone 1buten-3 one-1,1dimethyl Referencias 1. A. M. S. Silva, J. A. S. Cavaleiro, G. Tarrago and C. Marzin *New J. Chem.* 329- 335 (1999). 2. B. A. Bohm, in the *Flavonoids-Advances in Research Since 1986*, ed. J. B. Harborne, Chapman and Hall, London, 1994, pp 387-440. 3. A. Gadow, E. Joubert and C. F. Hansmann, *J. Agric. Food Chem.*, 1997, 45, 632. 4. M. A. Nazareno, A. M. Giannuzzo, H. T. Mishima, B. A. Lopez de Mishima. 51 st Annual ISE Meeting. Warsaw, Poland. September 2000. 5. P. Crsky, P. Zuman and V. Hork. *Collection Czechoslov. Chem. Commun.* 29, 4316 (1964) 6. E. P. Kohler, H. M. Chadwell. *Org. Syntheses*, Second Ed. Coll. Vol. I, p. 78. Wiley, New York 1947. 7. C. Iwakura, T. Abe, H. Inoue, *J. Electrochem. Soc.* 143 L.71 (1996). 8. H. Inoue, T. Abe, C. Iwakura, *Chem. Commun* 55 (1996) 9. *The Systematic Identification of Flavonoids*. T. J. Mabry, K. R. Markham and M. B. Thomas. Springer-Verlag. New York - Heidelberg. Berlin 1970. 226-250