NEW METHOD FOR ASSESSMENT OF HYDROGENATION CATALYSTS ACTIVITY BY ESTIMATION OF IMIDAZO[1,5-a]PYRIDINES REDUCTION TIME USING UV-SPECTROSCOPY

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Hydrogenation catalysts are widely used in modern fine organic synthesis for preparation of active pharmaceutical ingredients (API), compounds for agrochemistry, etc. Evaluation of such catalysts' activity is important task, because the properties of such systems strongly depend on their preparation method. Development of simple method for catalysts activity assessment is required for experiment planning as well as catalysts quality control.

The aim of this work was to develop a simple method for determination of hydrogenation catalysts activity. Hydrogenation of readily available imidazo[1,5-a]pyridines was chosen for this aim because of several reasons. 1. The reaction undergoes under 1 atm H_2 which excludes necessity of autoclave equipment. 2. Course of the reaction can be controlled by as simple method as UV-spectroscopy. There is significant difference in electronic spectra of the reaction products: λ_{max} = 365 nm for starting compound (A, Fig. 1), λ_{max} = 275 nm for the first hydrogenation product (B). These bands aren't obscured by adsorption of other compounds in the reaction mixture (B and C or A and C, respectively). λ_{max} = 220 nm for the second product (C), and the intensity of this peak can be calculated by analysis of total spectrum because of overlap with the bands of A and B in this spectral region.

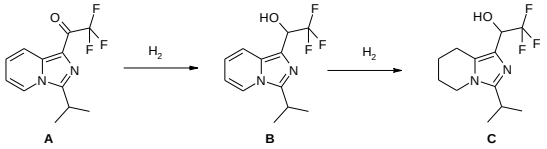

Fig. 1. Formulae of the compounds and reaction scheme

Hydrogenation of the substituted imidazo[1,5-a]pyridines was performed by gaseous H_2 at 20–40 °C and 1 atm. Formation of various products in certain time periods depended on the activity of the catalyst (Fig. 2), such dependency allowed to make conclusion regarding catalytic activity of the sample.

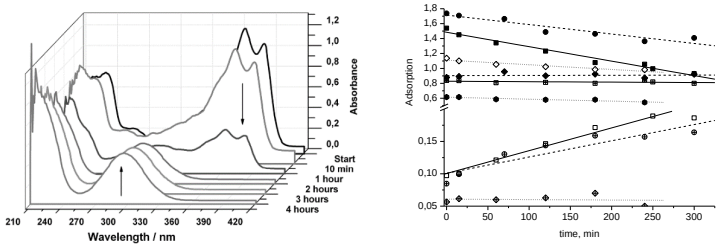

Fig. 2. Graphs, showing changes of spectra depending on hydrogenation time for different catalysts (one example on left; three different Pd/C samples on right)