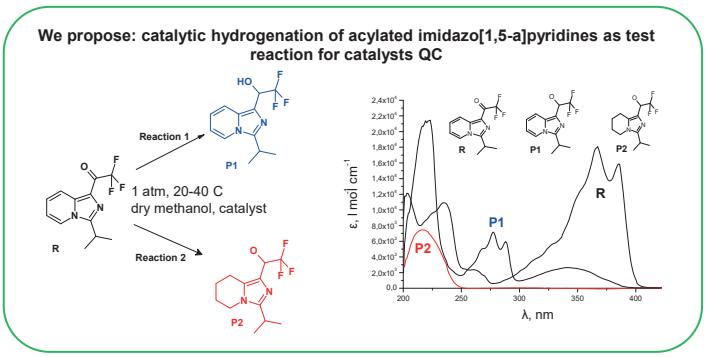
Quick evaluation of catalytic activity of hydrogenation cata-lysts by UV spectra using imidazo[1,5-a]pyridines as probes

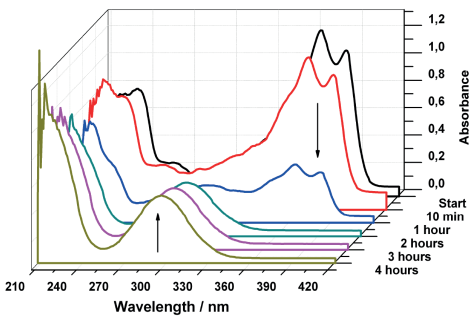
A. Lytvynenko, N. Ivanitsa, S. Sotnik, D. Tverdiy,
S. Ryabukhin, D. Volochnyuk, S. Kolotilov, A. Mishchenko

Introduction and Aim

Hydrogenation catalysts are widely used in modern organic synthesis and pharmacy for preparation of various compounds. Severe dependency of the catalyst's properties on preparation details is important problem for such catalysts application. The properties can significantly vary even between different batches of one supplier. Quick quality control (QC) is important task for hydrogenation catalysts application.

The aim of this work was to propose a method (test reaction) for hydrogenation catalysts QC, based on comparison of their catalytic activity in hydrogenation reactions that can be performed in mild conditions and does not require complicated products isolation and analysis.





The absorption bands of the products of partial or complete hydrogenation do not overlap. Formation of various products in certain time periods depends on the activity of the catalyst, such dependency allows to make conclusion regarding catalytic activity of the sample

Advantages of the method: pressure 1 atm; reaction monitoring by UV-spectroscopy with no need for products isolation; simultaneous formation of two products (P1 and P2) make an unique fingerprint of the catalyst

Comparison of catalysts activity in imidazopyridine hydrogenation (1 atm, 40 C) and in hydrogenation of quinoline to 1,2,3,4-tetrahydroquinoline (30 atm, 50 C)

Both in methanol; content of Pd in the reaction mixture is the same unless other is indicated

Catalyst	Pd content in the catalyst (%)	Decrease of reagent (R) concentration (n times) for 1 hour	Yield of tetrahydro-quinoline (%)	Specific surface, S_{BET} (m^2/g)	Gurvich pores volume (cm^3/g)	Dubinin-Radushkevich pores volume (cm^3/g)
C1	5	**123,6**	**73**	750	0,5668	0,2753
C2	10	1,515	12	**1215**	**0,9333**	**0,4091**
C3	5	7,114	25	700	0,5532	0,256
C4	10	2,787	36	870	0,5767	0,3209
C5	20	2,838	95*	623	0,2902	0,2262
Carbon	---	---	---	825	0,3952	0,3019

** 8-fold increase of Pd content in the reaction mixture*

The proposed method is suitable for express QC of the catalyst, which can be performed by comparison of the activity in the hydrogenation of imidazopyridine with reference sample. Formation of two different reaction products facilitates evaluation of the catalytic activity

There is no correlation between activity of the catalyst in hydrogenation of imidazopyridine and high-pressure hydrogenation of quinoline, so the method can't be used for evaluation of the catalytic activity in wide range of reactions without reference samples.

Notably, catalytic activity of the Pd composites does not correlate with Pd content. High surface area is also not the sole demand for high catalytic activity achievement. Pd particles size is the major factor which controls their catalytic activity in the reactions studied.

Contacts

Dmitriy M. Volochnyuk, Prof. Dr. Sci; Sergey V. Ryabukhin, Prof. Dr. Sci; Sergey V. Kolotilov, Prof. Dr. Sci.
s.v.ryabukhin@gmail.com, d,volochnyuk@gmail.com, s.v.kolotilov@gmail.com
Enamine Ltd, www.enamine.net
78 Chervonotkatska St, 02660 Kyiv, Ukraine