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TANDEM HYDROFORMYLATION-ACETALIZATION REACTION FOR CYCLIC ACETALS OBTAINING

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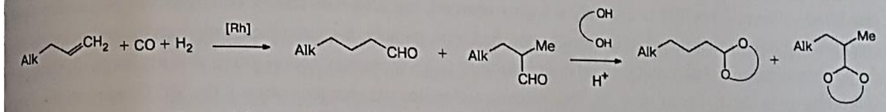
Gorbunov D.N.[1], *Kardasheva Y.S.*[1], *Maksimov A.L.*[1,2], *Karakhanov E.A.*[1].
[1]Lomonosov Moscow State University, Russia
[2]Topchiev Institute of Petrochemical Synthesis RAS
suskab_91@mail.ru

One of the green chemistry problems is to find the routes of polyols functionalization, which are by-products of biodiesel synthesis process (glycerol) and sugar industry waste (cyclic polyols). These raw materials can be refined to cyclic acetals by acetalization reaction with aldehydes. These acetals can be used as possible diesel additives [1]. In our work, we study how to obtain cyclic acetals by tandem ("one-pot") hydroformylation-acetalization reaction using olefins and polyols as raw materials:



We have already showed that cyclic acetals can be obtained with high yields in tandem reaction under homogenous catalysis conditions.

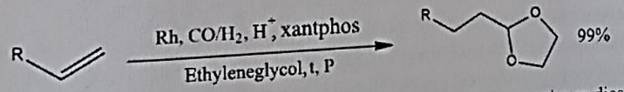
Polyol	Total acetals yield, %
Ethylene glycol	90
Glycerol	85
Xylitol	70
Sorbitol	70

Conditions: linear olefin C_6-C_{12}, Rh(acac)(CO)$_2$, toluene, H_2SO_4, p (CO/H_2)=4 MPa, 80°C, 5 h.

After this, we replaced homogenous sulfuric acid to heterogeneous solid acids (Wofatit, Ambelyst-16, KU-2) and also achieved high yields of acetals. In this case acid component of catalytic system can be easily separated and the corrosion of equipment can be prevent [2].

By using of water-soluble phosphine ligand TPPTS we showed that catalytic system containing Rh and sulfuric acid can be separated and reused without activity decrease.

Also we showed that linear acetal with 99% yield can be obtained in one step when steric phosphine ligand Xantphos is used:



In future, we plan to produce polyol acetals in large scale and investigate its properties as diesel fuel additives. We have already shown that 2% mass additive of acetals, produced from octene-1 and xylitol, increases lubricity of fuel by 30% and prevent formation of impurities in engine [3].

1. A.R. Trifoi, P. Ş. Agachib, T. Papa. *Renewable and Sustainable Energy Rev.* **62** (2016), 804.
2. D.N. Gorbunov, T.V. Shchukina, Yu.S. Kardasheva, N.A. Sinikova, A.L. Maksimov, E.A. Karakhanov. *Petroleum Chemistry*, **56** (2016), №8, 711.
3. D.N. Gorbunov, S.V. Egazaryants, Yu.S. Kardasheva, A.L. Maksimov, E.A. Karakhanov. *Russian Chemical Bulletin.* **55** (2015), №4, 943.

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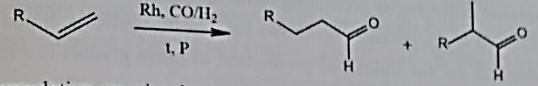
NEW HETEROGENEOUS HYBRID CATALYSTS FOR OLEFINS HYDROFORMYLATION

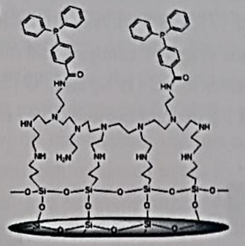
Safronova D.S., Gorbunov D.N, Kardasheva Y.S., Karakhanov E.A.
Lomonosov Moscow State University, Russia
SafDarya@mail.ru

Hydroformylation, or oxo-synthesis, is the main industrial way for aldehydes and alcohols producing.

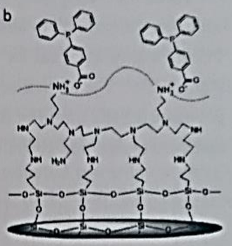


In terms of hydroformylation reaction investigationnew heterogeneous catalysts are being designedand synthesized. With use of supported catalysts, the problem of catalyst separation from reaction mix can be easily solved. In our work, we use known silica-supported NH_2-containaing material BP-1[1] for designing rhodium-containing catalysts. The first step of catalyst synthesis is NH_2-groups modification with phosphines. There are two main routes for this modification: by covalentor ionic coupling. So, we got precursors **a** and **b**.

a



b

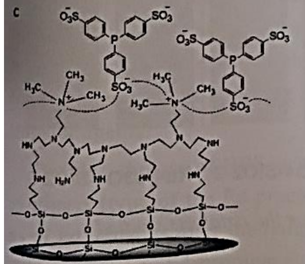


Then we obtained catalysts by interaction between these materials and Rh(acac)CO_2. Catalysts were characterized by solid-state NMR andatomic emission spectroscopy. Hydroformylation results achieved with catalysts are introduced in the table:

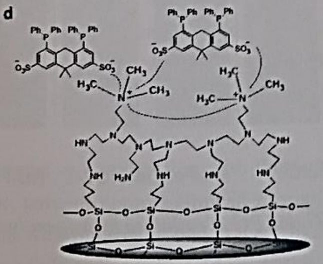
Catalyst	Conversion, %	Iso+hydr., %	Aldehydes, %	*n/i*
Rh-a	98	42	58	1.5
Rh-b	97	15	85	1

Conditions: octene-1, toluene, $p(CO/H_2)$=30 MPa, t=80°C, 5 hours.

c



d



Catalysts performhigh activity in octene-1 hydroformylation without activity loss after 5 runs.

Ionic coupling is easy and effective way for modification of NH_2-group containing supports. In the future, we planto get catalysts, based on modified materials of structure **c** and **d**.

1. J.Allen,E.Rosenberg,E.Karakhanov,S.Kardashev,A.MaximovandA.Zolotukhina.*Appl. Organometal. Chem.*25(2011), № 25, 245–254

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