

Referee comments to the master thesis entitled "Study of active centers in zeolite catalysts for cascade Prins-Friedel-Crafts reaction" written by Peter Golis:

The work is dealing with preparation and characterization of several micro- and mesoporous zeolites and use thereof as catalysts for Prins-Friedel-Crafts reaction (PFC). The zeolite series varies in T atom (Al, Ga), structural and textural properties and framework structure. Materials were characterized by many method commonly used for zeolitic species (XRD, FTIR, SEM,...) which showed the ability of Bc. Golis to analyze data. The Lewis and Bronsted acid sites concentration and were evaluated by FTIR using sorption of pyridine or or 2,6-ditertbutylpyridine. Finally, the catalytic properties in PFC reaction between butyraldehyde, 3-buten-1-ol and anisole was studied, while the author attempted to establish structure -activity correlations.

The work is clearly organized and written in good English, while minimum typos could be found. Conclusions are well supported by experiments.

I really appreciated the introduction part, which clearly and in detail explained not only catalytic materials studied but also methods used for their characterization. Unfortunately, the part dealing with well-characterized homogeneous catalyst for PFC was rather short. This could distinguish the unique role of Lewis and Bronsted sites in catalyzed PFC (conversion and selectivity). This refers to main problem of such complex heterogeneous systems for cascade reactions.

In summary, I recommend the thesis for defending and propose A class level (výborně)

#### Typos:

abstract (cz): replace ... a s také vysokým poměrem ... with a také s vysokým poměrem

list of abbreviations: Et<sub>3</sub>N<sup>+</sup> triethanol amine cation (Et is usually abbreviation for ethyl)

p. 27: ... form a homoallylic alcohol (such as **3**) - structure refers to acetal not alcohol

p.28: ... different homogeneous Bronsted (e.g. boron trifluoride etherate, ...) and Lewis (e.g. p-toluenesulfonic acid, ...) acid catalysis ... - I suppose that Bronsted and Lewis was interchanged (BF<sub>3</sub>.Et<sub>2</sub>O is not a Bronsted cat.)

#### Questions and comments:

1/FTIR spectra of Al-MFI and Ga-MFI showed an additional peak at ca 3500 cm<sup>-1</sup>. The peak position is close to a value characteristic for water, although, not observed in other samples. In addition, the peak does not move/hide after pyridine adsorption. Do you have any explanation of this peak origin?

2/ I miss comparison to homogeneous system (e.g. ScOTf<sub>3</sub>/TfOH - ref. 78), which showed significantly higher selectivity towards desired products (50-90%). It would be advantageous to perform at least one experiment for comparison.

3/ Recycling/reuse of catalyst is usually proposed as a strong advantage of heterogeneous catalysts in comparison to homogeneous one. Did you perform (or at least proposed) some recycling experiments, e.g., for most selective catalysts?

in Prague

23.5. 2022



Mgr. Jiří Pinkas, Ph.D.

Department of Molecular Electrochemistry and Catalysis

J. Heyrovský Institute of Physical Chemistry

Dolejškova 2155/3, 182 23, Prague 8, Czech Republic