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Scientific co-operation with professor Borowiecki♣

J. Ryczkowski
University of Maria Curie-Skłodowska, Department of Chemical Technology
pl. M. Curie-Sklodowskiej 3, 20-031 Lublin, Poland
e-mail: janusz.ryczkowski@umcs.eu

In present paper there will be discussed examples of author's scientific co-operation with professor Borowiecki. Generally all of them are from the area of heterogeneous catalysis with a feedback to chemical technology and an environmental protection.

1. INTRODUCTION

The impact of catalysis and catalysts is substantial. Today over 90% of all industrial chemicals are produced with the aid of catalysts [1,2]. Catalysts impact a sizable fraction of any nation's gross domestic product [2].

The story of catalysis has been told in the past by practitioners with different perspectives [2]. Lindstrom and Pettersson [3] chose to look at the development of catalysis over periods of time back to the dawn on civilization. This was the base of drawing scheme presented below (Figure 1) [4].

Taking into account published data [3,4], in present paper there will be discussed facts which took place in the 7th period of catalysis development.

2. BACKGROUND

The classical definition of chemistry is as follows: chemistry is the study of the composition and properties of matter, the transformations they undergo, and the associated energies. In this respect an applied chemistry is the application of the theories and principles of chemistry to practical purposes. All discussed

♣This article is dedicated to Professor Tadeusz Borowiecki on the occasion of his 65th birthday

examples fit well into this category [5-26]. As it is shown in Figure 2, all indicated sub-constituents are named in a classical way and cover broad areas of applied chemistry. Moreover, there is strong correlation between them.

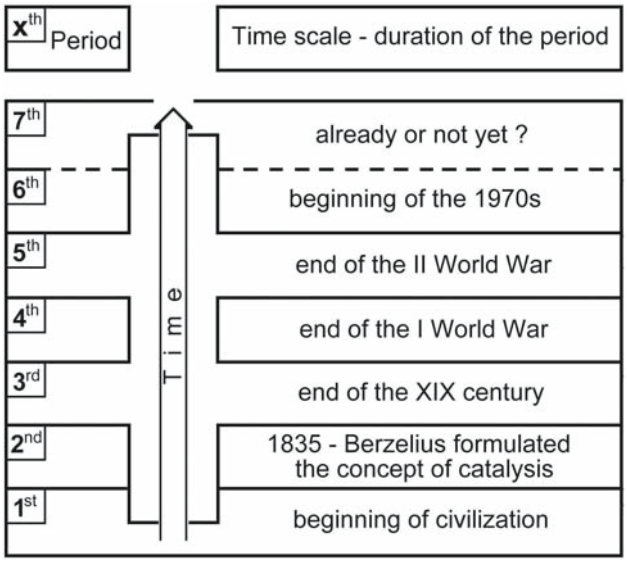

Fig. 1. Historical development of catalysis [4].

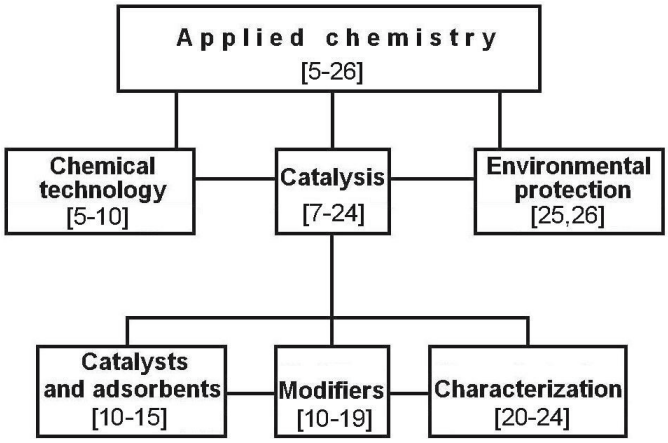

Fig. 2. Areas of author's scientific co-operation with professor Borowiecki – simplified scheme.

3. CHEMICAL TECHNOLOGY

A decade ago it was presented a simplified scheme covering preparation of catalyst with its characterization and testing [5]. However, it has to be extended to the form presented in Figure 3. Moreover, some new tendency in chemistry should be taken into account, i.e.: green chemistry. Green chemistry is a direction of chemical research and engineering that encourages the design of products and processes that minimize the use and generation of hazardous substances.

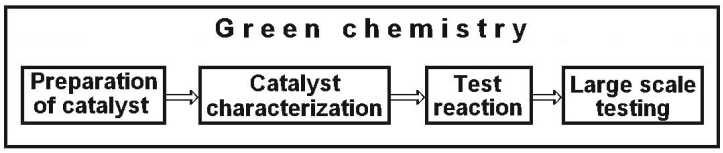

Fig. 3. Upgraded scheme of major directions in catalytic investigations.

Steam reforming (SR) of natural gas or higher hydrocarbons (HCs) conducted over nickel catalysts is the most popular method of synthesis gases and hydrogen production. The solutions of the problems appearing in the process of SR of HCs realization can be found in the way of the process organization modifications, changes in the reactor construction and/or the quality improvement of the applied catalysts.

Recently an essential modernization of SR due to the introduction of the new reactors solutions enabling the realization of integrated reforming has been achieved. The most relevant are two of them: AGHR (Advanced Gas Heated Reformer) and KRES (Kellogg Exchanger-Reformer System) [6].

Synetix (nowadays a division of Johnson Matthey) has developed a modified design of the GHR (Gas Heated Reformer) known as the Advanced GHR or AGHR (Figure 4) [27-29]. The AGHR design results in a reformer that is lower in cost, easier to operate, easier to fabricate and allows scale-up to capacities in excess of current world-scale throughputs.

AGHR has a small advantage on the technological side (smaller amount of the inert in the synthesis gas) [6].

The KAAP (Kellogg Advanced Ammonia Process) process is the first high-pressure ammonia synthesis process that makes ammonia from nitrogen and hydrogen without the aid of an iron-containing catalyst. The catalyst was developed by British Petroleum (BP) and contains ruthenium supported on carbon. The KRES provides the reformer gas for this process [28].

KRES is particularly attractive due to simple, reliable construction and the possibility of the process realization on a large scale.

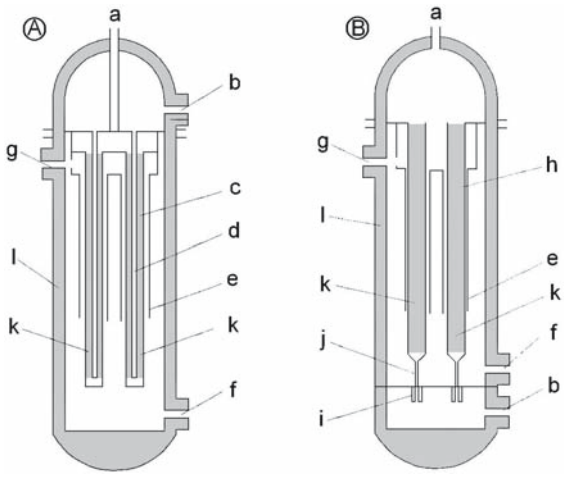

Fig. 4. Comparison of GHR (A) with AGHR (B): a – tube side inlet, b – tube side outlet, c – Scabbard tube, d – Bayonet tube, e – Sheath tube, f – shell side inlet, g – shell side outlet, h – catalyst tube, i – seal, j – tailpipe, k – catalyst, l – refractory lining [27].

The reviewed literature data were continued further with the extension to the processes of coal gasification for hydrogen production [7]. As an example a General Electric Global Research UFP (Unmixed Fuel Processor) technology was described. The UFP technology is a new, energy-efficient, and near-zero pollution concept for converting coal into separate streams of hydrogen, vitiated air, and sequestration-ready CO_2 (Figure 5) [30].

All the above mentioned literature studies [5-7] serve strong basis for the research which was conducted in the last three years [8-10, 31].

4. CATALYSIS

Catalysis was not invented. Actually, it is a phenomenon, which was observed and exploited long before the word was used to describe its underlying scientific reality (Figure 1). It is defined by the action of substance, which either promotes a particular reaction, or accelerates that reaction. Catalysis as a science has evolved along three major directions: preparation of catalyst, catalyst characterization and kinetics (test reaction) [5].

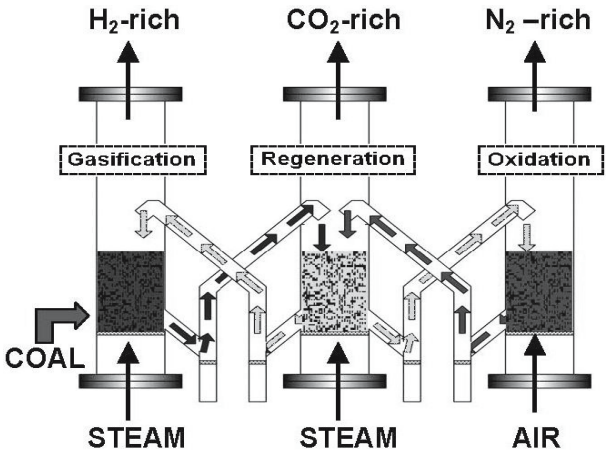

Fig. 5. Conceptual design of the UFP technology [7,30].

4.1. Catalysts and adsorbents

The activity and selectivity of a supported metal catalyst are strongly influenced by the amount of metal, the size of dispersed metal particles, the preparation method and the support composition. To improve the catalyst activity and its durability, it is necessary to obtain a well dispersed active phase in the catalyst.

In our laboratory an original technique of obtaining metal catalysts characterized by small metal crystallites, the so-called double impregnation method (DIM) was elaborated [32,33]. In contrast to the classical impregnation method (CIM), in the DIM preparation procedure the support is preliminary "activated" (modified) by EDTA (Figure 6).

This preparation procedure allows to obtain high dispersed and stable (after high temperature treatment) metal supported catalysts. Based on the conducted research it was concluded that changing the γ-alumina support do not has an influence on the impregnation mechanism by DIM (Figure 7).

Carbon dioxide hydrogenation is a perspective process from the point of practical application. Our studies were leaded over alkali metal-modified Ni/Al_2O_3 catalysts [13]. Different lithium containing compounds were used as modifiers (Figure 8).

We have concluded that the modification of Ni/Al_2O_3 catalysts with Li did not lead to any evident changes in selectivity towards CO and CH_4 in the CO_2 + H_2 reaction, whereas increasing temperature caused a decrease of selectivity towards methane [13].

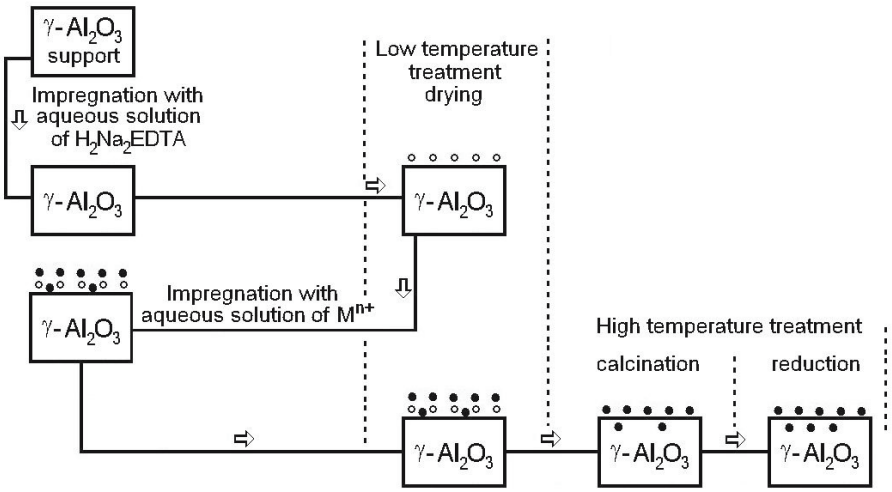

Fig. 6. Scheme of catalyst preparation by DIM (° – H_2Na_2EDTA, • – different M^{n+} and $M^{\delta+}$ species; where M is a metal) [5,11].

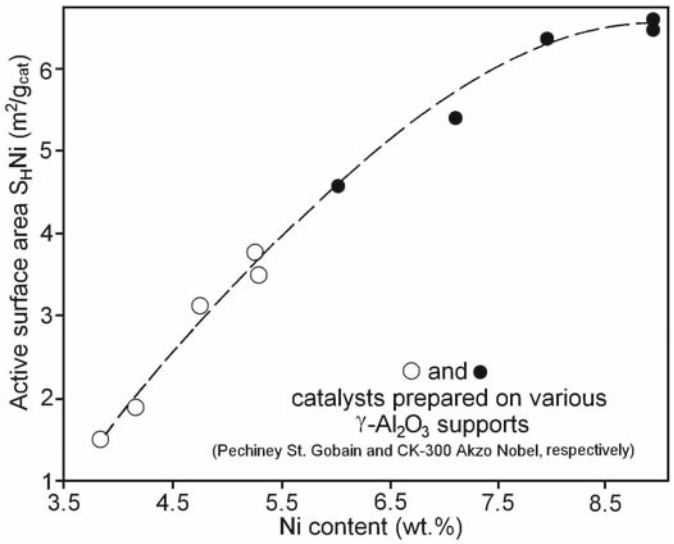

Fig. 7. Relationship between nickel content and active surface area [11].

The effect of molybdenum addition on the supported Ni-Mo catalyst reduction at the temperature >500°C for the systems in which the Ni:Mo > 5 was studied [14]. The results obtained show that reducibility of the catalysts is strongly influenced by small amount of Mo and decreases as Mo contents

increases. Decrease in reducibility is observed in the case of impregnated and co-precipitated nickel catalysts. The presence of water inhibits reduction process. Moreover, the higher oxidation temperatures increase interaction between Al_2O_3, NiO and MoO_3 [14].

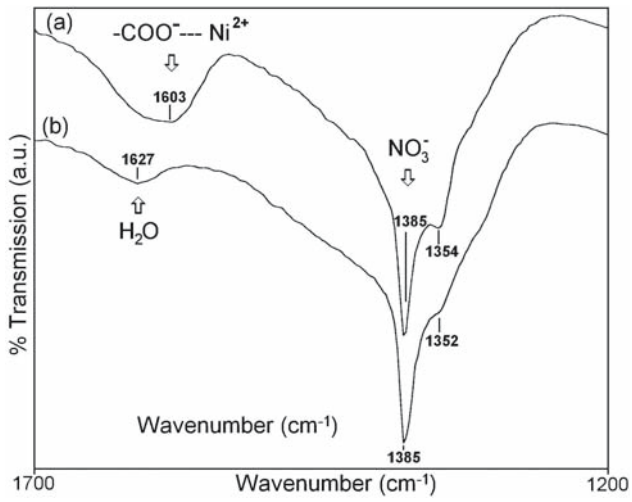

Fig. 8. FT-IR spectra of alumina support impregnated with an aqueous solution of H_2Li_2EDTA and subsequently treated with nickel nitrate solution: (a) after drying and (b) after drying and calcination [13].

Temperature removal of templating agent from MCM-41 silica materials was examined, among the others, by means of TPD (temperature programmed desorption), TPO (temperature programmed oxidation), MS (mass spectrometry) and FT-IR/PAS (Fourier transform infrared photoacoustic spectroscopy) [15].

The phenomena occurred during calcination of silicaeous materials of MCM-41 type in order to template removal in oxidative or inert gas atmosphere, in the region of low temperatures (<250^{o}C) are similar. Desorption of water, CO_2 as well as desorption and decomposition proceed in a similar ranges of temperature, and with a comparable intensity. Heating of the materials in higher temperatures causes the degradation of organic molecules. The presence of oxygen favors the different degradation processes, mainly oxidation to H_2O, CO_2 and NO_x. Thermal treatment in the inert gas atmosphere favors the processes of hydro-carbons break-up (gradual dehydrogenation). It is possible, that in the case of the inappropriate choosing of the parameters of the process the strong assembling of the coke remainders. The use of pure oxygen leads to more efficient removal of organics (Figure 9). The small coke residuals, removable above 600^{o}C, can

modify surface properties of the siliceous materials, what has an influence on their sorptive or catalytic properties.

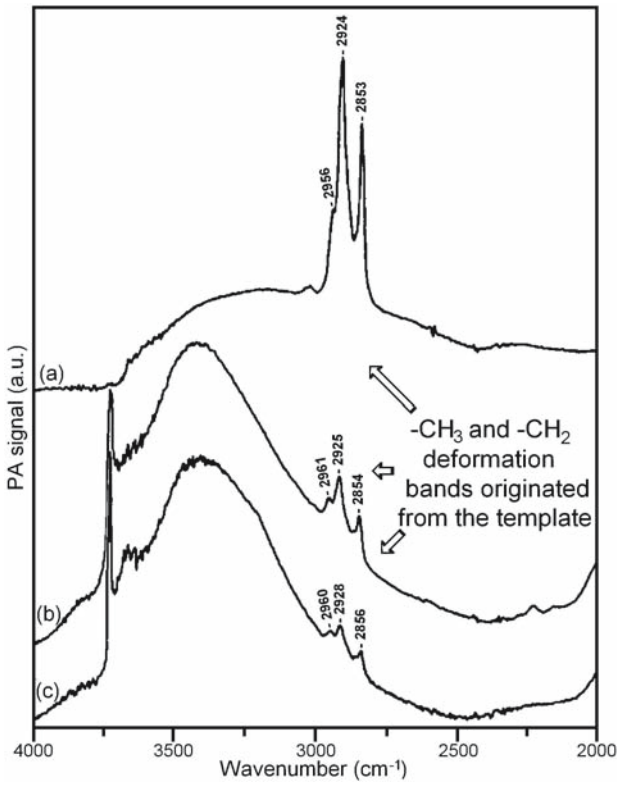

Fig. 9. FT-IR/PAS spectra of the studied MCM-41 samples after: (a) –synthesis, (b) – caliation, (c) – oxidation (calcination and oxidation were carried out at 500°C) [15].

Choosing the right conditions of the template removal are the key synthesis conditions having an influence on the final properties (chemical and textural) of mesoporous silica materials.

4.2. Modifiers

Simple and the cheapest way of the catalysts quality improvement is an introduction of promoters. It turns out, that the small amount of additives introduced into the catalysts' formula have a great influence on their textural properties, activity, selectivity and lifetime.

Promoters can be classed as substances which, when added to a catalyst as a minor component, improve one or more of the properties of the material with

respect to product formation. However, in the literature dealing with the catalytic problems there is no quantative determination of “small amount” or “minor component”. It seems, that the amount will vary with the catalyst (or reaction), and the precise determination of the standard value for the whole systems and processes is impossible [19]. Promoters belongs to the class of positive (+) modifiers (Figure 10).

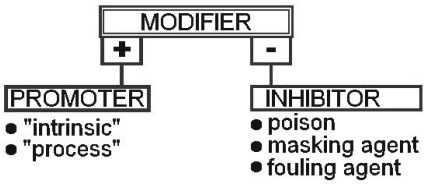

Fig. 10. Division of modifiers based on modifier action type [18,19].

In most cases promoters so-called “intrinsic” are introduced in to the catalyst during preparation procedures [11-19]. The modifier can have an effect on (Figure 11):

- the active phase,
- the support and the active phase-support interaction,
- the substrate and the catalytic reaction.

Modifiers are used for “tuning” all the useful properties of catalysts. The modifiers which prove efficient may be very different depending on reaction conditions, and they must be adapted for the given application.

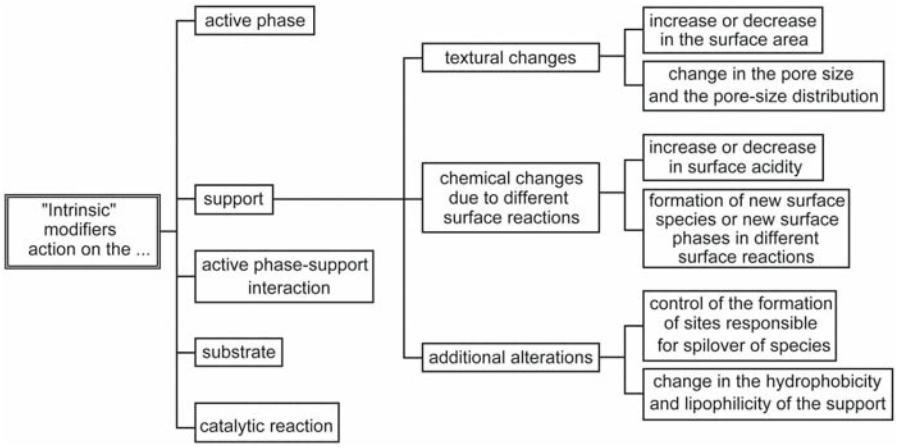

Fig. 11. Classification of modifiers action [16,34].

A condensed summary of the scientific activity in the area of modifiers application is given in Table 1.

Tab. 1. Examples of applied modifiers in our research [11-19].

Catalyst	Modifier	Comment	Reference
Ni/Al_2O_3	noble metals	catalytic studies	[10]
$Ni/\gamma\text{-}Al_2O_3$	H_2Na_2EDTA	adsorption, reduction properties	[11]
$Ni/\gamma\text{-}Al_2O_3$	hydroxy acids	FT-IR studies	[12]
Ni/Al_2O_3	lithium compounds	hydrogenation of CO_2	[13]
Ni/Al_2O_3	molybdenum	temperature programmed reduction	[14]
Ni/Al_2O_3	Sn, various organic compounds	FT-IR/PAS and catalytic studies	[16]
$Ni/\alpha\text{-}Al_2O_3$	K, Ba, Mo, W, Ce	physico-chemical characteristic and steam reforming of methane	[17]
Heterogeneous catalysts	different examples	short review	[18]
Promoters of the catalysts for CH_4 conversion into synthesis gases	various examples	chapter in the book, review with 165 references	[19]

In the following table (Table 2) there are data taken from the review mentioned already [19] but with the indication of the studies with the participation of professor Borowiecki.

4.3. Characterization

Physico-chemical characteristic of the prepared catalysts is an important stage for the correlation their properties with the catalytic results [20-24]. In the course of the catalytic reaction with the hydrocarbons or carbon oxides one of the problem of catalyst deactivation is carbon deposits formation. Induction period of coking, e.g. in the steam reforming of hydrocarbons, is an important parameter for the determination of the catalyst resistance for deactivation [20]. However, also the form of deposit formed is also important. The surface of the commercial catalyst KUB-3 for hydrogenation of benzene was studied by means of FT-IR/PAS [21].

Coke deposited on the mentioned catalyst has contained forms which were extractable with organic solvent in noticeable amount (Figure 12).

Tab. 2. Promoters of the catalysts for the steam reforming [19].

Kind of promoter	Action of promoter	Literature*
alkali - the most often potassium	● lowering of the coking rate ● loss of specific nickel activity ● escape possibility from the catalyst (corrosion hazard)	[35]
calcium oxide	● an increase in the thermal stability ● lowering of the coking rate	
barium oxide	● an increase in the thermal stability ● lowering of the coking rate	[36]
lanthanum oxide and oxides of rare earth metals	● an increase in the thermal stability ● an increase in the resistance resulting from the high-temperature influence of steam ● lowering of the increase of Ni crystallites and $NiAl_2O_4$ formation	[37]
cerium oxide	● an increase in the active surface area of Ni ● an increase in the resistance for sintering ● lowering of the coking rate (±) ● activity increase	[17]
molybdenum oxide	● difficulties in NiO reduction ● there is no influence on the active surface area ● noticeable limitation of the coking rate ● an extension of the coking induction period ● an increase in the activity at low amounts (~0.1 wt.%) of promoter ● an increase in the resistance on sulfur compounds poisoning	[38-40]
tungsten oxide	● difficulties in NiO reduction ● slight decrease in the nickel surface area ● lowering of the coking rate	[38, 41]
manganese oxide	● decrease in the nickel active surface area ● lowering of the coking rate (±)	[42]
vanadium oxide	● lowering of the coking rate	
chromium oxide	● lowering of the coking rate	
tin oxide	● lowering of the coking rate	[43]
copper, silver, gold	● lowering of the coking rate	[10]
precious metals (Rh, Ru, Pt)	● lowering of the coking rate ● activity increase	[10]

* paper/results co-authored by professor Borowiecki

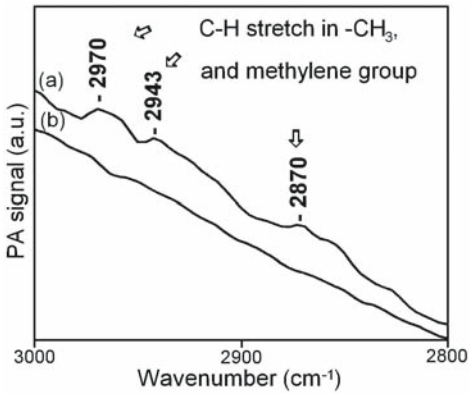

Fig. 12. FT-IR/PA spectra of the KUB-3 catalyst surface: (a) before, and (b) after extraction with dichloromethane [21].

In the following studies coked samples of KUB-3 catalyst taken from the different places of the industrial bed were examined by FT-IR and FT-IR/PAS [23]. Samples were extracted with CH_2Cl_2, and after organic solvent evaporation were subjected to IR analysis (Table 3).

Tab. 3. Position of the selected bands (cm^{-1}) in the examined extract samples [23].

Band position	Comment
3058 and 3030	C-H from aromatic ring and epoxy compounds, respectively
2920	CH_{2as} $-(CH_2)_n-$ (*as* – asymmetric stretching)
2850	CH_{2s} (*s* – symmetric stretching)
1713 and 1599	C=O
1447	$C-CH_3$ or $R-CH(CH_3)_2$ CH_{3as} – bending and CH_2 - scissoring
1375	$C-CH_3$ (e.g.,. near the carbonyl group)
1261 and 1090	C-O
1021	CH_3-O-C
890	ring vibrations of the epoxy compounds
802, 751 and 698	C-H

One very interesting observation has to be underline, namely: the KUB-3 catalyst was working in a reducing atmosphere. Some of the bands appeared in the recorded spectra can be attributed to the carbon-oxygen species. The explanation of this phenomena can be as follows: the reducing conditions during the hydrogenation of benzene to cyclohexane can also have an influence on unreduced NiO and/or alumina support. Probably during the long-lasting course of the conducted industrial process some amount of oxygen can be formed,

which subsequently interacts with the carbon species formed. This can be the source of carbon-oxygen compounds present in the studied samples [23].

Resistance to coking of the nickel catalysts is another example of the research conducted [24]. Carbon deposits formed on the examined samples were visualized by AFM (atomic force microscopy). It was concluded that deposits formed are typical for the steam reforming reaction of hydrocarbons. Moreover, steady-state rate of coking is determined by the active surface area of nickel.

5. ENVIRONMENTAL PROTECTION

Our scientific activity in this respect is mainly connected with the didactic process having students in an environmental protection studies [25, 26]. Environmental chemistry is the chemistry of the natural environment, and of pollutant chemicals in nature. On the other hand green chemistry seeks to reduce and prevent pollution at its source. However, there is strong correlation between both terms. Nowadays our research conducted is with the bases of green chemistry which has a direct impact on an environmental protection.

6. SUMMARY

Instead of a typical summary let me present a pictorial conclusion (Figure 13) based on the picture presented by A. Baiker (major directions of research and their interdependence) [44].

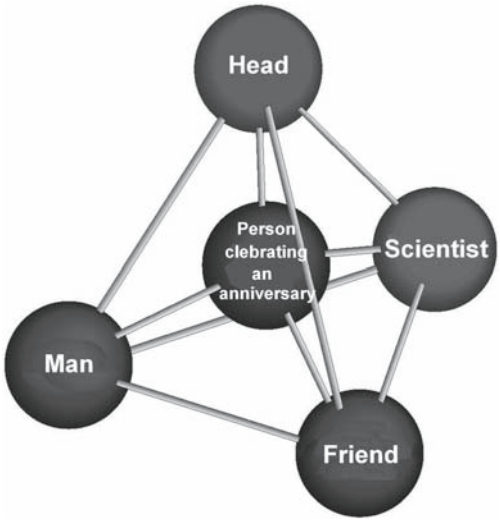

Fig. 13. Major directions of professor Borowiecki life activities and their interdependence.

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CURRICULUM VITAE

Janusz Ryczkowski. Born in Poland in 1959. Graduated from Nicolas Copernicus University in Toruń (1983). Received his Ph.D. and D.Sc. degree (1992 and 2004, respectively) in physical chemistry from the University of Maria Curie-Skłodowska in Lublin. Since 2007 university professor. Member of Polish Chemical Society (since 1984), Polish Catalysis Club (since 1993). Besides frequent short visits, he also made long-term stay to Central Research Institute of Chemistry of the Hungarian Academy of Sciences (Hungary) and Ecole National Superieure de Chimie de Lille (France).

Within the Socrates Programme (Teaching Staff Mobility) he has visited partner European universities with a series of lectures. Moreover, he is a Faculty representative in the European Chemistry Thematic Network Association. As for today he has been a reviewer of many papers submitted to leading scientific journals. On the request of the Authorities of Quaid-i-Azam and Punjab Universitie (Pakistan) since 1994 eighteen times he was a reviewer of doctoral theses. Moreover, three times he was a referee for a professor position. Seven times he was a reviewer of the Polish Ph.D. thesis. His main field of interest is preparation of supported catalysts, catalyst's modification, their characterization, and application of infrared spectroscopy (including photoacoustic) in catalytic research. He published over 130 papers.