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HYDROSILYLATION OF HYDROPHOBIC MATERIAL SUPPORTED BY PLATINUM CATALYSTS

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Abstract. Catalytic activity of platinum supported on styrene-divinylbenzene copolymer and fluorinated carbons was tested in reactions of hydrosilylation of several olefins and the way of platinum binding to the support surface as well as platinum oxidation states were determined. Catalysts based on the above hydrophobic materials make it possible to obtain high yields of desirable reaction products and in most cases the highest activity was shown by polymer-supported platinum catalyst. In addition to hydrophobicity, another factor appeared to influence catalytic activity, namely the kind of the catalyst precursor. It was established that some amount of unpolymerized vinyl groups that were present on the polymeric support surface was involved in the interaction with platinum. XPS spectra enabled to determine that platinum was present on the catalyst surface in 0 and +2 oxidation states, however Pt⁰ clearly predominated.

Key words: hydrosilylation, hydrophobic material, platinum catalysts, catalytic activity, polymeric.

1. Introduction

Hydrosilylation, i.e. the addition of Si-H bond to multiple bonds, is an important reaction from the viewpoint of both laboratory and commercial

scale applications. It was discovered in 1947 [13] and since then it arouses an unceasing interest of researchers that is reflected by thousands of papers published on this topic. However, a straight majority of them was devoted to hydrosilylation

in homogeneous systems, where transition metal complexes in solutions were employed as catalysts, or to hydrosilylation with the use of the mentioned complexes anchored to surfaces of inorganic or organic solids (hydrosilylation by heterogenized complexes) [8; 9]. Much less publications concerned hydrosilylation in typical heterogeneous systems, although the latter found an application to the commercial process of trichlorosilane addition to allyl chloride in the presence of active carbon-supported platinum catalyst [7]. However, in recent years an increase in the interest in heterogeneously catalyzed hydrosilylation has been observed [4; 11] because the latter offers a possibility of easier separation of catalysts from post-reaction mixtures thus facilitating catalyst reuse. The above advantage acquires a particular significance when the price of a catalyst is high and this is the case of catalysts for hydrosilylation processes since they are realized in the presence of costly transition metals. If hydrosilylation proceeds in a homogeneous system, the separation of a transition metal complex from reaction products and unreacted parent substances, e.g. by distillation, can be associated with its decomposition because many of these complexes undergo thermal degradation even below 150°C and other methods, e.g. extraction, usually result in a considerable loss of a precious metal [2]. Such a situation does not occur if the process is carried in heterogeneous system, because even in the case of a slight leaching of a noble metal, its loss is significantly smaller compared to that observed during attempts of catalyst recovery from homogeneous systems.

The choice of an appropriate support is of no less importance than that of active phase of a catalyst. We have focused our attention on the application of hydrophobic supports to prepare effective platinum catalysts for hydrosilylation since our preliminary experiments have shown that in a number of hydrosilylation reactions hydrophobic material-supported catalysts appeared to be superior to those based on hydrophilic supports such as alumina and silica. We have also aimed at selecting such supports which, in addition to their hydrophobicity, do not have acid centers on their surfaces and, due to this, they do not catalyze undesirable side reactions of isomerization. The supports selected for our study were styrene-divinylbenzene copolymer

(SDB) and fluorinated carbon (FC), because non-functionalized SDB is free of acid sites and surface acidity of FC is extremely weak ($H_0 \approx 9$) [3]. The performance of SDB- and FC-supported platinum catalysts was studied in several reactions of hydrosilylation.

2. Materials and methods

2.1. Preparation of catalysts

Supports employed for the preparation of catalysts were styrene-divinylbenzene copolymer (SDB) purchased from Aldrich and fluorinated carbons FC10, FC28 and FC65 (fluorine content: 10, 28 and 65 wt. %, respectively) obtained from Advanced Research Chemicals, Inc. (U.S.A.). Moreover, a commercial catalyst from Degussa – 1 % Pt supported on active carbon (Pt/C) was used for the sake of comparison. Surface areas of the supports, measured by low-temperature nitrogen adsorption on an ASAP 2010 sorptometer (Micromeritics) were: 1 172 m²/g for SDB and 178, 173 and 366 m²/g for FC10, FC28 and FC65, respectively. The supports were impregnated with chloroformic solution of platinum(II) acetylacetonate (from ABCR GmbH) or with a solution of hexachloroplatinic acid (from Aldrich) in 2-propanol to result in 1 wt. % Pt in a catalyst. Reduction of platinum catalyst precursors introduced onto surfaces of the above supports was performed at 160 °C in hydrogen flow. The polymer-supported catalyst prepared by using Pt(acac)₂ was labelled Pt/SDB, whereas that obtained by impregnation with H₂PtCl₆ was denoted by asterisk: Pt/SDB*.

2.2. Reactions studied

Reactions of hydrosilylation were carried out in the liquid phase in glass vials of 10 mL capacity. The vials were loaded with reacting substances and a catalyst taken in such amounts that the following ratio was met: $2 \cdot 10^{-4}$ mole Pt : 1 mole of a compound with Si-H bond : 1 mole of a compound with C=C bond. In the case of allyl polyether hydrosilylation with poly(hydromethyl-co-dimethyl)siloxane a greater amount of platinum ($5 \cdot 10^{-3}$ mole Pt) was used as well. Before the start of a reaction, each vial was sealed with a headspace aluminum cap and a teflon-lined septum followed by immersing into an oil thermostated

bath. Mixtures placed in the vials were stirred during reactions. All reactions were carried out at 100 °C, except for that between polyether and polysiloxane, where the reaction temperature was 125-130 °C due to problems with homogenization of the mixture. Post-reaction mixtures were analyzed on a gas chromatograph SRI 8610C equipped with a CP-Sil CB column (30 m). In the case of polyether – polysiloxane mixture, another method of evaluating conversion of reacting substance was applied, namely the determination of the loss of Si-H bonds on the basis of FT-IR spectra recorded on a *Bruker Tensor 27* Fourier transform spectrometer equipped with a SPECAC *Golden Gate* diamond ATR unit. The reason for replacing the GC analysis with infrared spectroscopic analysis was low vapor pressure of the polysiloxane, molecular weight of which was 5 500.

2.3. CP/MAS ¹³C NMR analysis

The measurements were performed on a 9.4 T Bruker DMX NMR spectrometer. Powdered sample was placed in a zirconia rotor of 4 mm outer diameter followed by spinning at the magic angle (54.44°) at 5 kHz. All spectra were recorded at 21 °C.

2.4. X-ray photoelectron spectroscopic (XPS) analysis

XPS spectra were recorded on a VSW spectrometer (Vacuum Systems Workshop Ltd., England) using a non-monochromatized Al K_α radiation (1486.6 eV). The X-ray gun was operated at 10 kV and 20 mA. The working pressure was 3 · 10⁻⁸ mbar.

2.5. Determination of platinum content in catalysts

To determine if leaching of platinum occurred during experiments with multiple use of catalyst, analyses for platinum content before and after using a catalyst in a hydrosilylation reaction were performed on an ICP-OES (inductively coupled plasma – optical emission) spectrometer (Vista MPX, Australia).

3. Results and discussion

3.1. Catalytic activity for hydrosilylation reactions

The choice of reactions to be carried out in the presence of hydrophobic material-supported platinum catalysts was based on the practical importance of reaction products. Epoxy functional silanes are among the most important adhesion promoters applied to create bonds between filler and polymer matrix thus improving physico-chemical and strength parameters of composites. Of practical importance are also epoxy functional siloxanes that are applied to the modification of epoxy resins thus making them more flexible, less susceptible to water sorption and more resistant to heat. Moreover, epoxy functional siloxanes find application in the manufacture of ionic silicone surfactants.

Octyl- and hexadecylsilanes are commonly applied as effective agents for hydrophobization and consolidation of building materials, architectural elements, monuments, etc. Alkylsiloxanes with long alkyl chain (at least 8 carbon atoms in alkyl group) are called silicone waxes which due to their moistening, softening, lubricating and spreading properties are widely applied in cosmetics, household chemistry as well as lubricating agents.

Silicone polyethers are important non-ionic surfactants that are also used in cosmetics and household chemistry. However, their most important application is the manufacture of polyurethane foams, both rigid and flexible ones. There are no substitutes for them and their role consists in the facilitation of mixing of foam components. They prevent from the formation of large bubbles, facilitate the control of fluidity of liquid mixture (that expands due to the bubble growth) and they enable accurate control of time and degree of foam opening (Fig. 1).

Measurements of catalytic activity for hydrosilylation of allyl glycidyl ether with

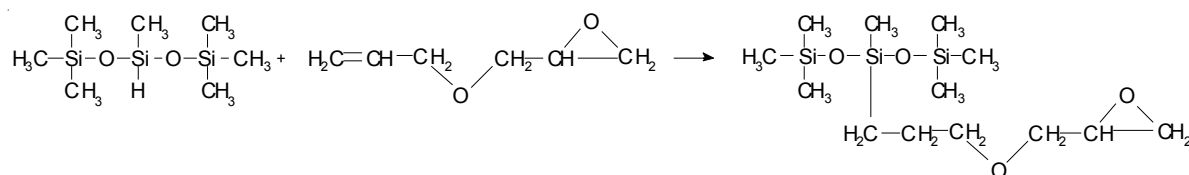


Fig. 1. Addition of 1,1,1,3,5,5,5-heptamethyltrisiloxane to allyl glycidyl ether

heptamethyltrisiloxane were conducted at 100 °C for 30, 60 and 180 minutes. Catalytic activity expressed as percent yield of desirable product, i.e. (3-glycidoxypropyl) bis (trimethylsiloxy) methylsilane, was presented in Fig. 2.

The yield of (3-glycidoxypropyl) bis (trimethylsiloxy) methylsilane after 3 hours from the beginning of the reaction carried out in the presence of polymer-supported platinum catalysts was well over 80 %, whereas in the case fluorinated carbon-supported ones it was considerably lower. The best of the latter catalysts (Pt/FC28) has reached the yield over 70 % only after 3 hours, while in the case of SDB-supported catalysts such an activity level was obtained already after 30 minutes (Fig. 2). The desirable product yield appeared to depend not only on the kind of support, but also on platinum precursor. The impregnation with platinum(II) acetylacetonate resulted in a more active catalyst than that prepared with the use of hexachloroplatinic acid. The activity of polymer-supported catalysts appeared to be clearly higher than that of the commercial catalyst. The selectivity to (3-glycidoxypropyl) bis(trimethylsiloxy)methylsilane was high (96-98 %) in the presence of all catalysts (Fig. 3).

Catalytic performance of polymer- and fluorinated carbon-supported platinum catalysts was determined at the same time intervals as in the case of hydrosilylation of the above ether with heptamethyltrisiloxane. Results of the measurements (shown in Fig. 4) point to high activity of both kinds of investigated catalysts which after 3 hours reaches the level of 87 % in the presence of all catalysts, except for that supported on fluorinated carbon with the lowest fluorine content (FC10). However, even in the latter case the yield of desirable product, i.e. 3-glycidoxypropyltriethoxysilane, exceeded 80 %.

The activity of the reference (Pt/C) catalyst after 3 hours was similar to that of catalysts prepared by us, but it reached the level of 86 % as early as after 30 minutes. All the catalysts studied were characterized by a high selectivity to the desirable product (≥95 %) (Fig. 5).

Both SDB-supported catalysts made it possible to obtain good yields of desirable reaction product (1-hexadecyltriethoxysilane), but similarly as it was in the case of allyl glycidyl ether hydrosilylation with heptamethyltrisiloxane, results shown in Table 1 point to platinum(II) acetylacetonate as a better platinum catalyst precursor than H₂PtCl₆. Also FC-supported

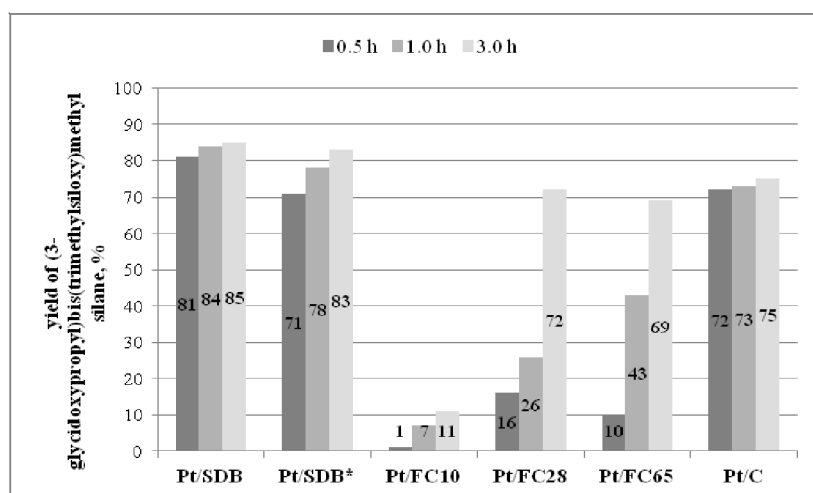


Fig. 2. Activity of polymer- and carbon-supported platinum catalysts for hydrosilylation of allyl glycidyl ether with heptamethyltrisiloxane at 100 °C

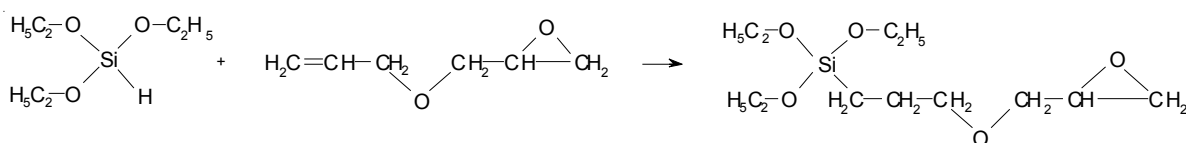


Fig. 3. Addition of triethoxysilane to allyl glycidyl ether

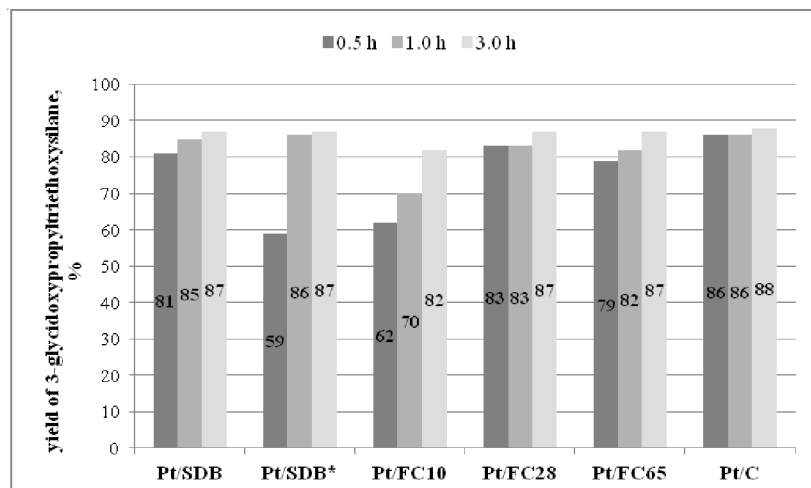


Fig. 4. Yields of 3-glycidoxypropyltriethoxysilane obtained in the reaction between triethoxysilane and allyl glycidyl ether proceeding in the presence of SDB- and FC-supported platinum catalysts

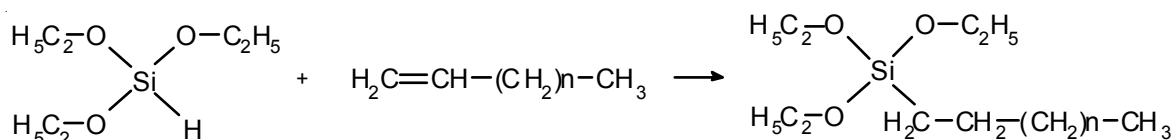


Fig. 5. Addition of triethoxysilane to 1-hexadecene and 1-octene:
 n=13 or 5 in the case of the addition to 1-hexadecene and 1-octene, respectively

Table 1

Yield and selectivity to the desirable reaction product and conversion of parent substances in the process of 1-hexadecene hydrosilylation with triethoxysilane carried out at 100 °C on polymer-supported platinum catalysts

Reaction time, h	Yield of desirable reaction product, %	Selectivity to desirable reaction product, %	Conversion degree of triethoxysilane, %	Conversion degree of hexadecene, %
Pt/SDB				
0.5	84	96	86	87
1	84	96	86	87
3	86	96	88	87
Pt/SDB*				
3	74	97	78	79

catalysts have shown a very good performance in the discussed reaction that was particularly impressive (91 %) in the presence of Pt/FC28 (Fig. 6).

Hydrosilylation of 1-octene with triethoxysilane on polymer-supported catalysts (Table 2) proceeded, to a considerable extent, in the way similar to that of hexadecene. Platinum(II) acetylacetonate as a platinum catalyst precursor appeared to be again a more advantageous choice.

However, somewhat unexpected results were obtained while using FC-supported catalysts (Fig. 7). The Pt/FC10 catalyst, the performance of which in the reactions described previously was

poorer than that of other fluorinated carbon-supported catalysts, this time appeared to be the most active, surpassing even polymer-supported catalysts. Water contact angle for hydrophobic materials should be greater than 90° and in the case of FC10 it is a bit below the above value, namely it equals to 85°, whereas for FC28 to 120° [8] (for SDB copolymer it is in the range of 109-117° [10]) and this fact brings into conclusion that support hydrophobicity, although it plays an important role in hydrosilylation reactions, is not the only factor affecting the activity of catalysts for hydrosilylation processes. It is worth

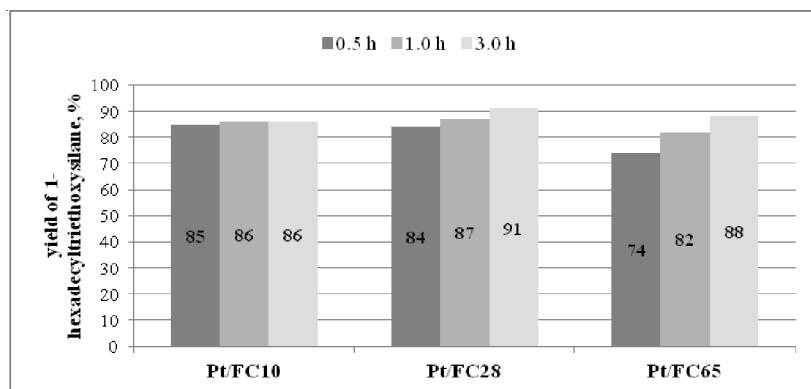


Fig. 6. Catalytic performance of the catalysts studied for the addition of triethoxysilane to 1-hexadecene at 100 °C

Table 2

Yield and selectivity to 1-octyltriethoxysilane and conversion of parent substances in the process of triethoxysilane addition to 1-octene carried out at 100 °C on polymer-supported platinum catalysts

Reaction time, h	Yield of desirable reaction product, %	Selectivity to desirable reaction product, %	Conversion degree of triethoxysilane, %	Conversion degree of octene, %
Pt/SDB				
0.5	69	95	75	73
1	83	95	85	86
3	86	95	87	86
Pt/SDB*				
3	67	94	83	82

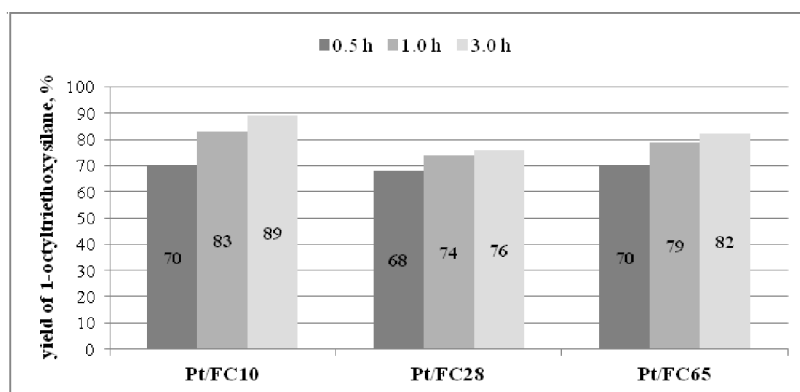


Fig. 7. Catalytic activity for 1-octene hydrosilylation with triethoxysilane at 100 °C in the presence of Pt supported on fluorinated carbon of different fluorine content

mentioning that the catalytic performance of platinum supported on fluorinated carbon containing 65 % F, which is characterized by the highest water contact angle (125° [8]) from among supports employed in our study, is not the best catalyst among them.

As it was already mentioned, hydrosilylation in heterogeneous systems facilitates catalyst

reuse. This is why we have undertaken tests for multiple use of catalysts in the reactions of hydrosilylation of 1-hexadecene and 1-octene with triethoxysilane (Fig. 8).

It results from Fig. 8 that Pt/SDB catalyst is characterized by a high activity that remains on a constant level for three reaction runs and then declines, most likely due to platinum

leaching from the catalyst as can be concluded from ICP-OES analyses for platinum content in catalysts after their use in the fifth run of reactions between TES and hexadecene (0.40 % Pt) as well as TES and octene (0.44 % Pt) (Fig. 9).

First measurements of catalytic activity for the discussed reaction were performed at the mole ratio of platinum to parent substances (polysiloxane and polyether) equal to $2 \cdot 10^{-4}$ and resulted in Si-H conversion of 73 % and 77 % after 2.5 and 5 hours, respectively, of conducting the reaction. Then the ratio was increased to $5 \cdot 10^{-3}$ in hope for raising the conversion. Si-H conversion observed at the latter ratio after 5 h of the reaction was 80 % and after 10 and 15 h it was 82 and 86 %, respectively. The obtained results show that in the presence of Pt/SDB catalyst it is possible to reach a high Si-H conversion already at the ratio of Pt to polyether and polysiloxane equal to $2 \cdot 10^{-4}$ and a further increase in the ratio seems pointless taking into account the cost of platinum-containing catalyst.

3.2. Platinum species on catalyst surfaces

In the previous section we have shown that platinum supported on styrene-divinylbenzene copolymer makes an effective

catalyst for hydrosilylation. Now we should answer the question: how platinum is bound to the support surface? X-ray diffraction and hydrogen chemisorption measurements [6; 16] showed the presence of a considerable number of large Pt crystallites which are weakly bound to the support surface and thus are vulnerable to leaching. However, on SDB surface, there are also sites capable of interacting with platinum in a stronger way. Potential centers for the interaction between the metal and the support are vinyl groups, certain number of which (rather small one) could remain after the polymerization process. Such a hypothesis was put forward in our earlier paper [6] and in the present study we verified this conjecture by recording solid-state ^{13}C NMR spectra of SDB support and Pt/SDB catalyst (Fig. 10). Such measurements enabled to determine a possible loss of signal originated from carbon atoms present in $-\text{CH}=\text{CH}_2$ group.

The studied system is simple for analyzing because the signal coming from double-bonded carbon atoms of vinyl groups appears at about 112 ppm and the signal ascribed to carbon atoms of benzene ring is located at about 127 ppm. Areas under each peak were measured and ratios of peak areas originating from carbon atoms of vinyl groups and those of aromatic rings were

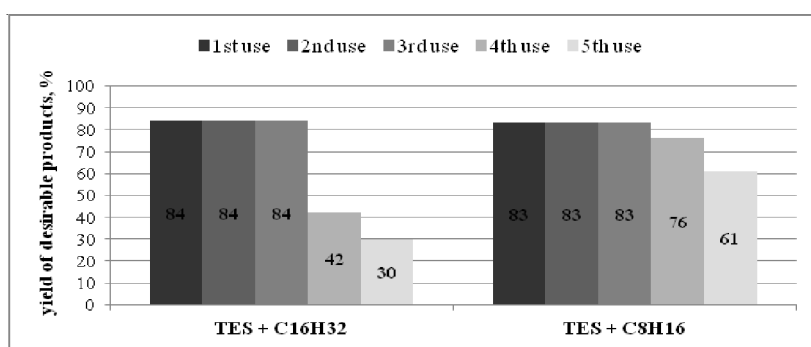


Fig. 8. Yields of desirable products (hexadecyltriethoxysilane and octyltriethoxysilane, respectively) obtained after 1h in tests for multiple use of Pt/SDB catalyst in hexadecene and octene hydrosilylation with triethoxysilane (TES) at 100 °C

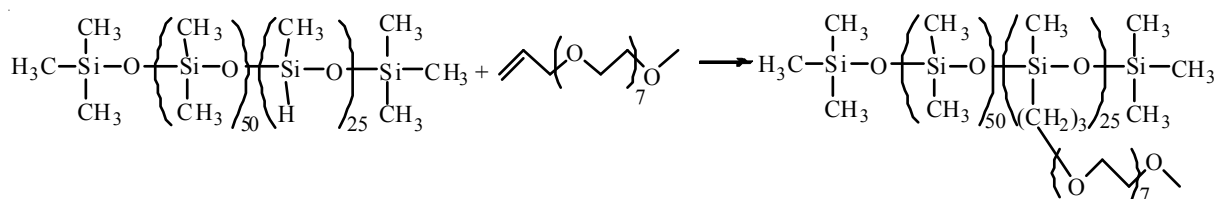


Fig. 9. Addition of poly(hydromethyl-co-dimethyl)siloxane to allyl polyether

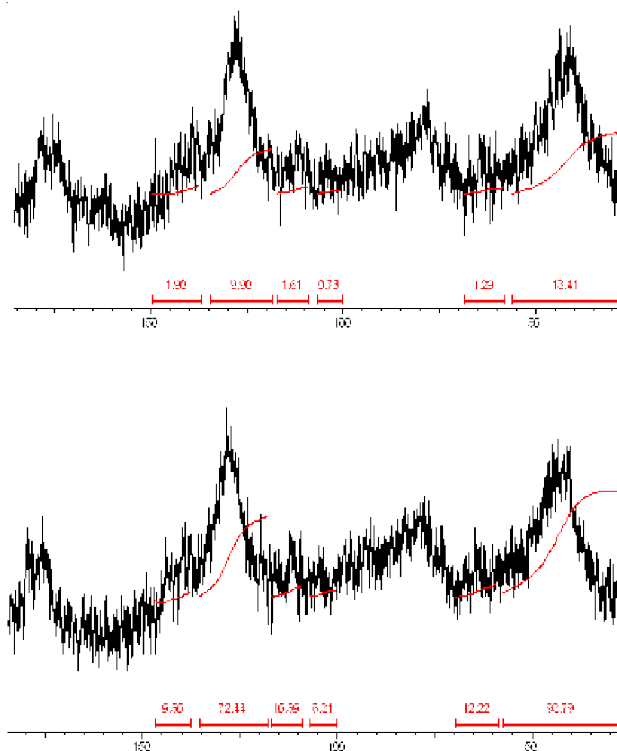


Fig. 10. ¹³C NMR spectra of SDB support and Pt/SDB catalyst

calculated. Results of the calculations are shown in Table 3.

Table 3
Changes in the number of unpolymerized vinyl groups in SDB occurring as a result of introducing platinum onto the support

Sample	-CH=CH ₂	-CH=CH-	(1)/(2)
	(1)	(2)	
SDB	1.61	9.90	0.16
Pt/SDB	10.39	72.44	0.14

Data presented in Table 3 show that the introduction of platinum onto the support causes a small decrease in the number of double bonds originating from free vinyl groups. This result supports the hypothesis presented in Ref. [6] that a surface complex was formed by the interaction between platinum and vinyl ligands.

Next question concerns the oxidation state of platinum in Pt/SDB catalysts. Admittedly, X-ray diffraction pattern presented in Ref. [16] clearly indicated the presence of Pt⁰, but small amounts of other platinum species (undetected by X-ray technique) possibly can also exist on

the catalyst surface. Temperature-programmed reduction profile of SDB-supported Pt(acac)₂ contained two peaks, the first of which corresponded to the reaction of hydrogen with platinum(II) acetylacetonate and the second one to the reaction between hydrogen and products of partial decomposition of Pt(acac)₂ [16]. X-ray photoelectron spectroscopy (XPS) measurements carried out in the present study have shown the presence of platinum species in the +2 oxidation state, in addition to those in zero oxidation state (Table 4).

Table 4
Results of XPS analysis of Pt/SDB and Pt/SDB* catalysts

Band	Peak maximum, eV	Concentration in the sample, %	Quantitative ratio	Ascription
Pt/SDB				
Pt 4f _{7/2}	72.2	0.60	83	Pt ⁰
	75.1	0.10	17	Pt ²⁺
Pt/SDB*				
Pt 4f _{7/2}	73.0	0.70	73	Pt ⁰
	75.3	0.25	27	Pt ²⁺

The spectrum of Pt/SDB catalyst was deconvoluted into two components at 72.2 and 75.1 eV (Fig. 11). Peak corresponding to binding energy of 72.2 eV can be ascribed to Pt⁰, whereas

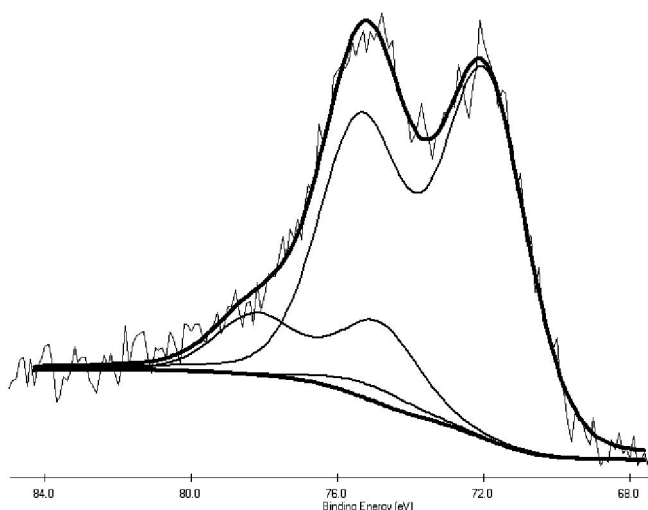


Fig. 11. The Pt 4f XPS spectrum of Pt/SDB catalyst

that at 75.1 eV to Pt⁺² [10; 17]. The deconvolution of Pt/SDB* spectrum resulted in two components at 73.3 and 75.3 eV ascribed to Pt⁰ and Pt⁺², respectively (Table 4).

Results of XPS analysis bring into conclusion that reduction of platinum precursors at 160 °C leads to a clear predominance of Pt⁰, although some amount of Pt⁺² remains on the catalyst surface. This is in agreement with data obtained earlier from XRD and TPR H₂ analyses. It is possible that both Pt⁰ and Pt⁺² take part in catalyzing hydrosilylation reactions, however, the main contribution to catalytic process seems to come from Pt⁰ which predominates on surfaces studied.

4. Conclusions

Hydrophobicity of catalyst support, although very important to many hydrosilylation reactions carried out in heterogeneous systems, is not the only factor deciding of the performance of supported platinum catalysts for hydrosilylation. Results obtained by using different platinum compounds for the preparation of catalysts for hydrosilylation show that the kind of catalyst precursor also belongs to the factors influencing catalytic activity.

In addition to high activity, the studied catalysts are highly selective. One of reasons for their high selectivity is the absence of acid centers on surfaces of styrene-divinylbenzene copolymer and fluorinated carbons, therefore

undesirable side reactions of isomerization do not proceed.

Tests for multiple use of catalysts for hydrosilylation of hexadecene and octene with triethoxysilane have shown that yields of desirable products are maintained on a high constant level for three runs and then they decline due to platinum leaching from the catalysts.

The presence of a certain amount of unpolymerized vinyl groups on polymeric support surface results in their interaction with platinum and the complex formed between them plays a role in binding platinum to the support surface.

Platinum is present on the polymeric support surface in 0 and +2 oxidation states and the former of the oxidation states clearly predominates.

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ГИДРОСИЛИЦИРОВАНИЕ ГИДРОФОБНЫХ МАТЕРИАЛОВ В ПРИСУТСТВИИ ПЛАТИНОВЫХ КАТАЛИЗАТОРОВ

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Аннотация. Катализаторы на основе платины для гидрофобных материалов позволяют получать высокие показатели желательных продуктов реакции. Помимо гидрофобности появился еще один фактор влияния на каталитическую активность, а именно вид катализатора-предшественника. Установлено, что некоторое количество неполимеризованных виниловых групп, которые присутствовали на полимерной опорной поверхности, были вовлечены во взаимодействие с платиной.

Ключевые слова: гидросилицирование, гидрофобный материал, платиновые катализаторы, каталитическая активность, полимерный.