

O₂ and O₃ regeneration of PtReSn/Al₂O₃ and PtReGe/Al₂O₃ naphtha reforming catalysts prepared by catalytic reduction

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ABSTRACT

The deactivation and regeneration of naphtha reforming PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalysts prepared by catalytic reduction were studied. The extent and nature of coke deposition as determined by TPO were related to catalyst properties such as dispersion, acidity and Cl content. The PtReSn catalyst was the most resistant to coke deactivation.

Regeneration was performed by calcination in oxygen at 450 °C or ozone at 125 °C at variable regeneration times. Regenerated catalysts were evaluated by cyclopentane hydrogenolysis, cyclohexane dehydrogenation and n-heptane reaction tests. Regeneration by oxygen burning-off was the most effective for decoking. However, oxygen combustion produced more segregation of the metal function than ozone regeneration. With the regeneration conditions used in this work, the original acidity of the catalysts cannot be recovered.

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1. Introduction

Deactivation occurring during the catalytic reforming of straight-run naphtha makes it necessary to regenerate the catalyst by a continuous or periodic combustion of the formed coke deposits. The main parameters affecting the formation of coke are the partial pressure of hydrogen and hydrocarbon, the space velocity, the reaction temperature and the composition of the feedstock [1]. The coking rate can vary widely depending on the kind of catalyst, i.e. the kind of metal, the size of the metal particles, the promoters and the support [2].

In the bifunctional catalysts, the properties of the metal function are specially responsible for the formation of coke [1] because: (i) the olefins are formed on metal and can be polymerized over the acid sites of the catalyst; (ii) the polymers formed on the support can be stabilized by dehydrogenation through inverse spillover of hydrogen; (iii) coke precursors can be destroyed on the metal at high temperatures or by hydrogen spillover.

During coke burning-off, the exothermal effects are the key ones to control. The active metal component is mainly sintered during coke combustion due to the great temperature increase (>500 °C). Due to the high temperatures the regeneration in the presence of oxygen can cause metal sintering problems and segregation of the metals which compose the metal function [3]. For this reason a

chlorine/water injection step is necessary for obtaining catalysts with high metal dispersion. Fung et al. [4] pointed out that chlorination is a necessary step in order to achieve a high dispersion of Pt supported on zeolites after regeneration with O₂/N₂ mixtures. However in the case of the Pt–Ir/Al₂O₃ catalyst the Cl content of the support is a very important factor for recovering the dispersion of supported Ir [5]. In the case of the regeneration using ozone, the temperature of combustion can be low due to the high oxidizing activity of this compound. At high temperatures, ozone can be decomposed in the gas phase, without reacting with the coke on the catalyst [6]. In order to prevent the segregation of the metal phase we have used ozone to eliminate the coke in a previous work dealing with regeneration of naphtha reforming catalysts [7]. The cost of implementing an ozone-based regenerating technology is much higher than that needed when oxygen is used as oxidant.

In the present work, the deactivation and regeneration of PtReGe/Al₂O₃ and PtReSn/Al₂O₃ naphtha reforming catalysts were studied. The catalysts were prepared by catalytic reduction.

2. Experimental

2.1. Preparation of the Pt–Re/γ–Al₂O₃ base catalyst

γ–Al₂O₃ (Cyanamid Ketjen CK-300, ground and sieved to 35–80 meshes, pore volume = 0.5 cm³ g^{−1}, specific surface area = 180 m² g^{−1}) was used as support and calcined at 500 °C for 4 h in air in order to eliminate organic impurities. HCl (0.2 M) was added (1.5 cm³ g^{−1}) and the slurry was let to rest for 1 h.

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H_2PtCl_6 and NH_4ReO_4 were added and the slurry was gently stirred for 1 h at room temperature. It was slowly dried at 70°C until a dry powder was obtained which was further dried overnight at 120°C . Finally the powder was calcined in air ($60\text{ cm}^3\text{ min}^{-1}$, 300°C) and reduced with H_2 ($60\text{ cm}^3\text{ min}^{-1}$, 500°C). The final content of Pt and Re on the catalyst was 0.3 wt.% and 0.3 wt.%, respectively. This catalyst is named Pt–Re base catalyst.

2.2. Preparation of trimetallic catalysts

Trimetallic PtReGe/ Al_2O_3 and PtReSn/ Al_2O_3 catalysts were prepared by catalytic reduction by making use of the catalytic action of the metal function of the bimetallic parent catalyst (Pt–Re). The bimetallic catalyst was first reduced at 500°C for 1 h in hydrogen ($60\text{ cm}^3\text{ min}^{-1}$) and then cooled down to room temperature while maintaining the hydrogen flow. Then 25 cm^3 of a degassed GeCl_4 or SnCl_2 solution with the appropriate concentration were poured in the reactor. After 1 h of contact with the catalyst under constant hydrogen flow ($300\text{ cm}^3\text{ min}^{-1}$), the solution was drained. Then the catalyst was dried at 100°C under hydrogen overnight. Finally the catalyst was activated by reduction in hydrogen at 500°C for 2 h.

2.3. Measurement of the Pt, Re, Sn, Ge and Cl contents

The composition of the metal function was determined by inductively coupled plasma-optical emission spectroscopy (ICP-OES) after digestion in an acid solution and dilution. The chlorine content of the catalysts in their final state, i.e. after activation, was determined by means of potentiometry. All these analysis were carried out by the CNRS at the Service Central d'Analyse (Vernaison, France).

2.4. Cyclopentane hydrogenolysis (CP)

Before the reaction the catalysts were reduced for 1 h at 500°C in H_2 ($60\text{ cm}^3\text{ min}^{-1}$). Then they were cooled in H_2 to the reaction temperature (350°C). The other reaction conditions were: catalyst mass = 150 mg, pressure = 0.1 MPa, H_2 flow rate = $40\text{ cm}^3\text{ min}^{-1}$, cyclopentane flow rate = $0.483\text{ cm}^3\text{ h}^{-1}$. The reaction products were analyzed in a gas chromatograph connected on-line.

2.5. Cyclohexane dehydrogenation (CH)

The reaction was performed in a glass reactor with the following conditions: catalyst mass = 50 mg, temperature = 300°C , pressure = 0.1 MPa, H_2 flow rate = $36\text{ cm}^3\text{ min}^{-1}$, cyclohexane flow rate = $0.727\text{ cm}^3\text{ h}^{-1}$. Before the reaction was started, the catalysts were treated in H_2 ($60\text{ cm}^3\text{ min}^{-1}$, 500°C , 1 h). The reaction products were analyzed in a gas chromatograph connected on-line.

2.6. Dynamic chemisorption of CO

This technique was used to measure the dispersion of the Pt metal particles on the surface of the catalyst. Calibrated pulses of the adsorbate were injected in a stream of inert gas that flowed over the sample. These pulses were sent to the reactor until the sample was saturated. At the beginning of the experiment the sample (400 mg) was reduced at 500°C ($10^\circ\text{C min}^{-1}$) for 1 h. Then nitrogen was made to flow over the sample for 1 h at 500°C in order to eliminate adsorbed hydrogen. Then the sample was cooled down to room temperature in nitrogen and pulses of $0.6\text{ }\mu\text{mole}$ of CO were sent to the reactor.

2.7. Isomerization of 3,3-dimethyl-1-butene

This reaction was used to selectively and quantitatively determine the concentration of the Brønsted sites of the catalysts. The

skeletal isomerization of 3,3-dimethylbut-1-ene (33DMB1) is relatively simple with only two main products: 2,3-dimethylbut-1-ene (23DMB1) and 2,3-dimethylbut-2-ene (23DMB2). This reaction consists in sigmatropic shift of the methyl group in the carbocation and can proceed only when Brønsted acid sites are present at the catalyst surface. The molecule does not contain hydrogen atoms in the α position in respect to the double bond and therefore cannot form allylic species and isomerizes according to this reaction pathway. It may be therefore used as a probe reaction to measure the concentration of acid sites [8]. 100 mg of sample were first loaded in a quartz reactor. Then the sample was reduced in situ for 1 h at 500°C (H_2 flow = $60\text{ cm}^3\text{ min}^{-1}$) and cooled down to 300°C in nitrogen. Then the flow was switched to air ($30\text{ cm}^3\text{ min}^{-1}$) and the catalyst was calcined at this temperature for 1 h. The reaction was performed at 250°C , 0.1 MPa, with a reactant flow rate of 15.2 mmol h^{-1} and a reactant partial pressure of 20 kPa.

2.8. n-C₇ reaction test

It was performed in a tubular fixed bed reactor, at atmospheric pressure, 500°C , $\text{H}_2/\text{n-C}_7$ (molar) = 6, WHSV = 4 h^{-1} . The catalyst was previously reduced in H_2 ($12\text{ cm}^3\text{ min}^{-1}$) for 1 h at 500°C . The analysis and assessment of the reaction products were done by chromatographic analysis using a ZB-1 capillary column and a flame ionization detector.

2.9. Deactivation

1 g of the catalyst was placed in the fixed bed reactor and then reduced in H_2 ($30\text{ cm}^3\text{ min}^{-1}$, 500°C , 1 h). Then cyclopentane was fed at $1.2\text{ cm}^3\text{ h}^{-1}$ to the reactor maintained at 500°C in flowing hydrogen ($30\text{ cm}^3\text{ min}^{-1}$) for 7 h.

2.10. Temperature programmed oxidation

Carbon deposits formed on the surface of the catalysts during reaction were studied by temperature programmed oxidation (TPO). 40–60 mg of the coked catalyst were first charged in a quartz micro reactor. Then the carbon was burned in an oxidizing stream ($40\text{ cm}^3\text{ min}^{-1}$ of diluted oxygen, 5% O_2 in N_2). The temperature of the cell was increased from 30°C to 700°C with a heating rate of $10^\circ\text{C min}^{-1}$. The outlet gases were fed to a methanation reactor where CO_2 and CO were quantitatively transformed into CH_4 over a Ni/Kieselgur catalyst in the presence of H_2 . The $\text{N}_2:\text{CH}_4$ stream was connected to a flame ionization detector (FID) and the signal produced was continuously recorded in a computer. The carbon concentration of the catalysts was calculated from the area of the TPO trace (FID signal as a function of the temperature of the cell) by reference to calibration experiments performed with catalysts with known carbon concentration.

2.11. Regeneration with O₃

It was performed in a fixed bed reactor maintained at 125°C . The reactor was heated to the target temperature in air ($40\text{ cm}^3\text{ min}^{-1}$). Then the gas was changed to ozone. The time of regeneration used was 0.5, 1 and 2 h. The samples used (500 mg) were previously deactivated as indicated above.

2.12. Regeneration with O₂/N₂

The samples (500 mg) previously coked were regenerated in a fixed bed reactor at 450°C , which was heated from room temperature at $10^\circ\text{C min}^{-1}$. The sample was first heated in nitrogen until the target temperature was reached. Then the gas flow was changed

Table 1
Values of carbon content after deactivation, percentage of conversion of cyclohexane and cyclopentane, amount of chemisorbed CO, chlorine content, total Brønsted acidity and selectivity to 23DMB1 and 23DMB2 of the fresh PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalysts.

Catalyst	C (%)	CH (%) ^a	CP (%) ^b	CO/Pt	Cl (%)	Isomerization of 3,3-dimethyl-1-butene		
						Brønsted acidity ^c	23DMB1 ^d	23DMB2 ^d
PtReGe	0.56	23.4	7.1	0.45	1.10	0.71	36	64
PtReSn	0.48	32.0	8.9	0.34	0.97	0.60	32	68

^a Cyclohexane conversion.

^b Cyclopentane conversion.

^c Conversion of 3,3-dimethyl-1-butene (relative to the base PtRe/Al₂O₃ catalyst).

^d Selectivity to 23DMB1 and 23DMB2 obtained in the reaction of 3,3-dimethyl-1-butene.

to a O₂/N₂ mixture (5% O₂ 40 cm³ min⁻¹). This regeneration treatment was performed for 0.5, 2 and 4 h.

3. Results and discussion

The metal content of the Pt–Re base catalysts, as determined by ICP-OES, was 0.22 wt.% of Pt and 0.23 wt.% Re while the content of Sn or Ge added by catalytic reduction were 0.1 wt.% for each one.

Table 1 summarizes the values of carbon content obtained after the test of deactivation as well as the activity of the metal and acid functions of the PtReGe/Al₂O₃ and PtReSn/Al₂O₃ fresh catalysts.

It has been demonstrated that the amount of coke deposited on the metal depends on its dispersion. It has been reported that the coking is lower on the metal function if small Pt particles are present [9–11]. It has also been found that the metal dispersion has an influence on the total amount of coke deposited (over the metal and acid functions) because higher coke amounts have been found on catalysts with higher metal dispersions [12]. The reactions of hydrogenolysis and coking are structure-sensitive reactions [13] and both require the same type of metal sites [14]. Coking also occurs over the support (acid function) where polymerization reactions take place [15,16].

It can be seen in Table 1 that the percentages of carbon on the PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalysts after deactivation are 0.56 and 0.48, respectively. This means that the PtReSn catalyst is more resistant to deactivation by coking than the PtReGe one. The higher stability can be explained from the various results shown in Table 1. The catalyst containing Sn has a higher dehydrogenating activity than the one with Ge in spite of its lower capacity for chemisorbing CO. This seemingly contradictory result can be explained by considering that a solid solution is formed between Pt and Ge that is superficially enriched in Pt atoms. In this way the metal particles might chemisorb CO but not hydrogen. Wootsch et al. [17] have found that adding 1–2 monolayers of Ge caused a new type of interaction in the sites containing both Pt and Ge. These sites adsorbed CO but did not adsorb hydrogen [17]. A solid solution of Pt–Ge might have been formed thus creating “bulk bimetallic catalysts” with more surface Pt atoms not interacting with Ge. In previous works we have reported for PtReSn/Al₂O₃ catalysts with various Sn contents that a direct correlation exists between the decrease of the dehydrogenating activity, as measured by the cyclohexane test reaction, and the chemisorption of CO. As the Sn content was increased, the activity was proportionally affected [18]. An analogous effect occurs with Ge doped catalysts but no linear correlation between the dehydrogenating activity and CO chemisorption capacity can be found [19]. The hydrogen chemisorption of the catalysts was not determined because it does not reflect the real dispersion value. For example, for Pt–Re/Al₂O₃ catalysts it was demonstrated that hydrogen chemisorption techniques give lower dispersion values due to an electronic effect of Re over Pt or by dilution of Pt ensembles by Re atoms. This effect produces a decrease in the number of pairs of neighboring Pt atoms

capable of chemisorb hydrogen [20,21]. It is expected that the effect of Ge and Sn increases the hydrogen chemisorption problems.

It is well known that dehydrogenation reactions can proceed on simple (monoatomic) sites, but hydrogenolysis requires catalytic sites of a more complicated morphology (clusters or ensembles) [22–24]. The addition of an inactive metal (Sn, Ge) to the catalyst places “spacers” between Pt–Re groups and reduces the effective size of the catalytically active Pt–Re ensembles, hindering hydrogenolysis processes. It can be seen in Table 1 that the hydrogenolytic capacity of the Pt–Re ensembles is more affected by Ge addition than by Sn addition. The lower coke deposition on the PtReSn/Al₂O₃ trimetallic catalyst can be explained by considering its greater hydrogenolytic capacity (for destroying coke precursors) and its lower amount of Brønsted acid sites. It is important to comment that in our experimental conditions the skeletal isomerization of 3,3-dimethylbut-1-ene (33DMB1) produces only two products: 2,3-dimethylbut-1-ene (23DMB1) and 2,3-dimethylbut-2-ene (23DMB2), and as it can be seen in Table 1, the selectivity is similar for both catalysts. The dehydrogenation reaction, according to the results found, is less affected. This confirms that the polymerization of the coke deposits is controlled by the strength of the acid function of the catalyst.

The location of the coke (on the metal or the support) and its nature (soft or graphitic) are more important parameters for the catalytic stability than the amount of coke itself [1]. The combustion of the coke deposits on the metal sites occurs at low temperatures (250–300 °C) while the coke on the support is burned-off at high temperatures (400–450 °C) [25]. For catalysts coked in the laboratory, these burning ranges are displaced to lower temperatures. Sachtler et al. [26] have reported that chlorine modifies the siting of the coke deposits (i.e. on support or metal). In the presence of chlorine, the percentage of coke deposited on the support is increased to the detriment of that on the metal function [26]. This phenomenon is confirmed by comparing the TPO traces of the non-regenerated PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalysts, which possess a Cl content of 1.10 and 0.97%, respectively, which show that a majority of coke is deposited on the support (see Fig. 1). The little amount of coke on the metal function is also due to the presence of metallic ensembles constituting of three metals, thus hindering the coke deposition. The combustion peak on coked PtReGe/Al₂O₃ at 120 °C is due to the elimination of hydrocarbons adsorbed during the cyclopentane coking reaction.

Fig. 1 shows also the TPO traces of the regenerated PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalysts. They were regenerated in O₂ or O₃ for 0.5 and 2 h. At low regeneration times (0.5 h), the remaining coke induces no TPO peak at low temperatures with O₂. This means that only the coke deposited on the support remains. When the TPO traces of the non-regenerated and the ozone-regenerated PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalysts are compared, it can be seen that the burning pattern is drastically changed as the time of regeneration is increased. For a 2 h treatment with ozone both catalysts display a combustion zone at 200–300 °C. This pattern change is due to the formation of carbon oxidized species during the com-

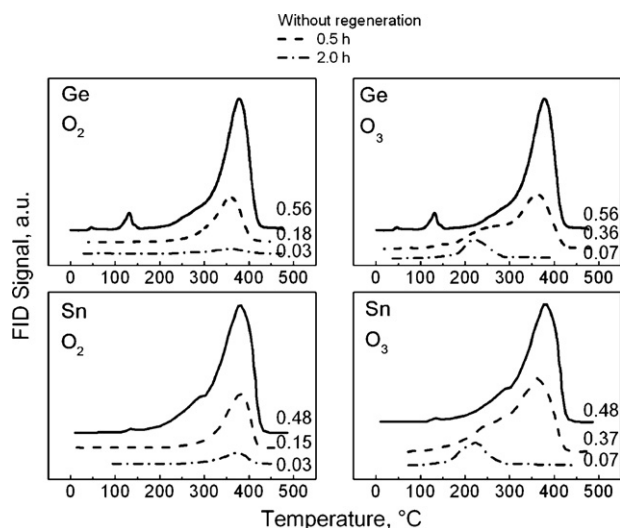


Fig. 1. TPO traces of the PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalysts (noted Ge and Sn, respectively). Coked samples (non-regenerated) and samples regenerated with O₂ and O₃ for 0.5 and 2 h.

bustion with ozone. These species can be burned with ozone at lower temperatures than with oxygen during the TPO test [27].

Table 2 shows the percentage of carbon remaining on the coked PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalysts partially regenerated with oxygen and ozone. The table also contains the amount of carbon eliminated during the regeneration. According to these results the regeneration in oxygen turns out to be more convenient for removing coke.

When using O₂ for regeneration, the percentage of eliminated coke as a function of time of regeneration is practically the same for both catalysts. This indicates that the coke has the same reactivity towards oxygen whatever the catalyst and that the phenomenon is independent of the amount of coke present. When regenerating with ozone, at low regeneration times (0.5 and 1 h) the combustion is more effective on the PtReGe/Al₂O₃ catalyst while at high regeneration times the amount of combusted coke is the same for both catalysts. The differences in the combustion between O₂ and O₃ may be due to the fact that at 450 °C (temperature for regeneration in O₂) oxygen is so reactive that differences in the surface nature of the support cannot influence the combustion process.

The results of cyclohexane conversion on the deactivated and regenerated PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalysts are plotted in Fig. 2 as a function of the percentage of eliminated coke. The activity in dehydrogenation of cyclohexane of both coked catalysts was null. It can be seen that as the coke content decreases the conversion is increased. This is a natural tendency if we consider that

Table 2

Amount of carbon on the coked PtReGe/Al₂O₃ and PtReSn/Al₂O₃ catalyst as a function of the oxidizing medium and the time of regeneration. Regeneration in O₂ at 450 °C and in O₃ at 125 °C.

Catalyst	Regeneration time (h)	Carbon (%)		Eliminated carbon (%)	
		O ₂	O ₃	O ₂	O ₃
PtReGe	0.5	0.18	0.36	67.9	35.7
	1.0	–	0.17	–	69.6
	2.0	0.03	0.07	94.6	87.5
	4.0	0.01	–	98.2	–
	PtReSn	0.5	0.15	0.37	68.8
PtReSn	1.0	–	0.23	–	52.1
	2.0	0.03	0.07	93.8	85.4
	4.0	0.01	–	97.9	–

(–) Experiments not performed.

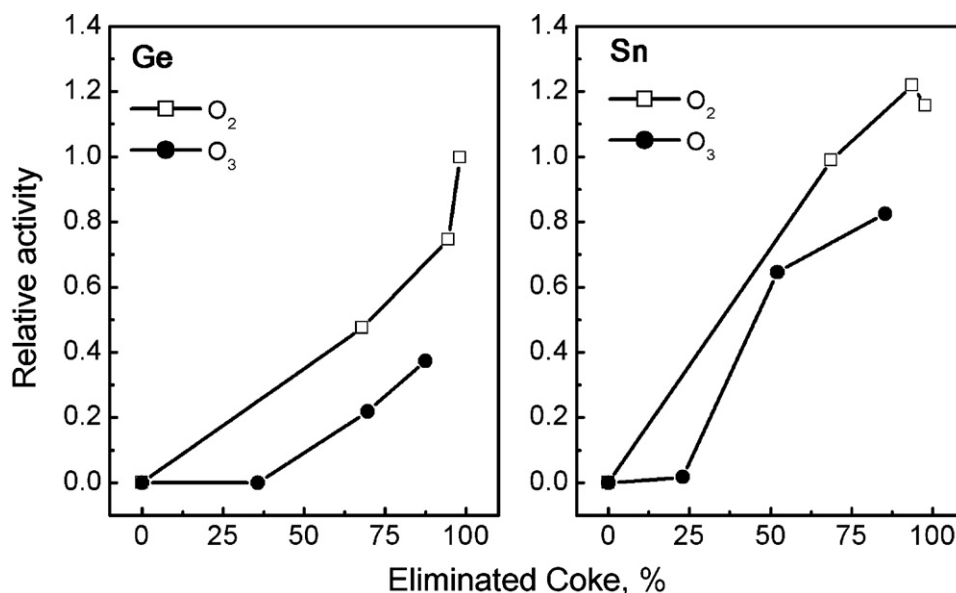


Fig. 2. Relative activity in cyclohexane dehydrogenation referred to fresh catalyst of each series. Samples of variable coke content regenerated with O₂ and O₃.

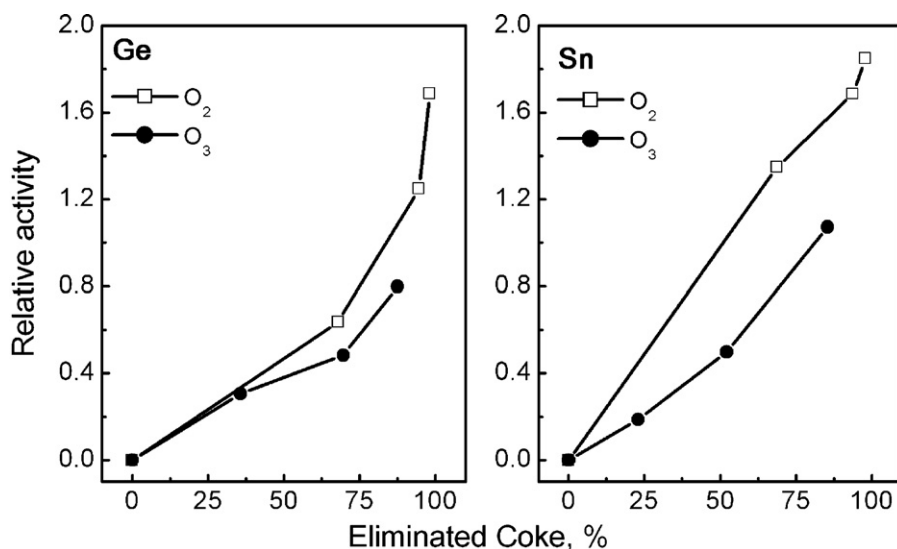


Fig. 3. Relative activity in cyclopentane hydrogenolysis at 5 min time-on-stream referred to fresh catalyst of each series. Samples of variable coke content regenerated with O_2 and O_3 .

at lower coke contents the fraction of accessible metal atoms is higher. On both catalysts, the combustion with oxygen turned out to be more effective for recovering the activity of the metal function.

These results are in accord with those of the TPO tests. These tests showed that the coke burning-off occurred preferentially on the metal function when oxygen is used for the combustion. However, the elimination of coke with O_2 produces a segregation of Sn and Ge from the active metal phase and for this reason the regenerated catalysts have a higher metal activity than the fresh ones (relative activity higher than 1). The PtReSn/ Al_2O_3 catalyst suffered a higher segregation than the PtReGe/ Al_2O_3 one. If regenerated with ozone, the activity of the metal function is not fully recovered, because only 85% of the coke is burned.

The reaction of hydrogenolysis is an undesired reaction in naphtha reforming but is a very useful test reaction for evaluating the interaction between the metals that compose the metal function. For example, it can be used to determine whether Ge and Sn are in interaction or not with the active phase. A high hydrogenolytic activity indicates that Ge and Sn are segregated. Fig. 3 shows the values of cyclopentane conversion for the coked and partially regenerated catalysts. It can be seen that the recovery of the hydrogenolytic activity is higher when the regeneration is performed with oxygen. The higher activity of the regenerated catalysts (i.e. relative activity higher than 1) can be explained by assuming that regeneration produces metal oxides that segregate with no possible co-reduction during the reduction step in order to restore interaction.

Menon [28] has proposed a classification of reactions as coke-sensitive or coke-insensitive. In the case of a coke-sensitive reaction, the coke deposits over the active sites produce a decrease in the activity. Hydrogenolysis is one of coke-sensitive reactions [2]. According to this classification, the structure and location of the coke have a more important influence than its amount. According to this theory, the recovery of the hydrogenolytic activity would be achieved only when all the coke on the metal is removed. This is fulfilled in the case of the oxygen regenerated catalysts. The TPO tests indicated in this case that all the coke on the metal function had been eliminated.

The studied catalysts had a little amount of coke on their metal function. The elimination of this deposit is supposed to occur in a short time and therefore the original dehydrogenating and hydrogenolytic activity should be restored after a 30 min burning-

off treatment. However the results show an increase of the activity in both reactions as the coke is gradually eliminated. This can be due to two reasons: (a) coke burning-off is not selective; (b) the inactive metals (Ge and Sn) are segregated during regeneration. The lack of selectivity for burning-off the coke can be related to the real maximum combustion temperature (as determined by the maximum in the TPO trace) that is lower than the temperature used in the oxygen regeneration tests ($450^\circ C$). This means that the regeneration was carried out at a temperature at which any kind of coke can be burned-off. The segregation of Sn or Ge is clear from the results of the dehydrogenation and hydrogenolytic tests, which showed that regenerated catalysts were more active than the fresh ones. The segregation occurring during the oxygen regeneration is higher than that produced by the regeneration with ozone.

The n-heptane test reaction allows one to determine if an adequate balance between the metal and acid functions exists. Fig. 4 shows the conversion of n-C₇ as a function of the regeneration time. Fresh and regenerated (2 h in oxygen or ozone) catalysts are used. It can be seen that in the case of the regenerated catalysts, independently of the treatment (oxygen or ozone) the activity level of

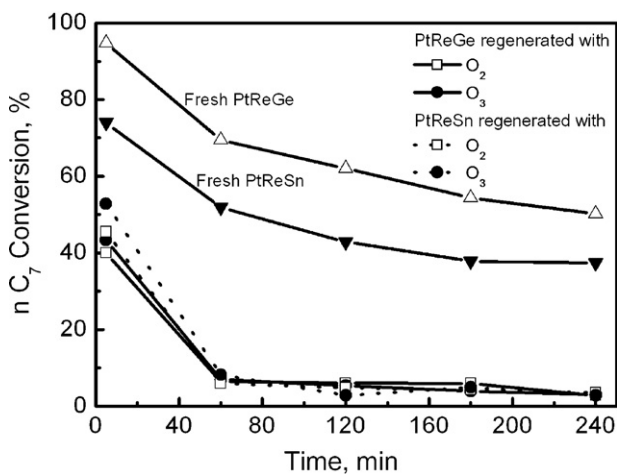


Fig. 4. n-C₇ conversion as a function of time for PtReSn/ Al_2O_3 and PtReGe/ Al_2O_3 catalysts. Fresh samples (noted Sn and Ge) and coked samples after regeneration in O_2 or O_3 for 2 h.

the fresh catalysts cannot be restored. Moreover regenerated catalysts get drastically deactivated. This great activity decline was addressed to the loss of chlorine during regeneration.

4. Conclusions

The results of the cyclopentane coking tests indicate that the PtReSn/Al₂O₃ catalyst is more stable than the PtReGe/Al₂O₃ one. This is attributed to its higher capacity to destroy coke precursors and its lower concentration of Brønsted acid sites.

The regeneration of both catalysts with O₂ produces similar coke elimination as a function of time whatever the catalyst while the ozone-assisted coke combustion is more effective in the case of the PtReGe/Al₂O₃ sample at low regeneration time.

After regeneration under O₂, only the coke on the support remains whereas after 2 h of treatment under O₃, the combustion peak at high temperature has completely disappeared.

The catalytic activity measurements (dehydrogenation and hydrogenolysis reaction tests) indicate that the non-active metals of the metal function are segregated from the metal particles to the support during the regeneration step. This segregation is more important in the case of the regeneration with O₂.

Finally the results obtained in the n-C₇ reaction test indicate that both kinds of regenerating treatment cannot restore the original acid properties of the evaluated catalysts.

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