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Characterization of Residual Coke during Burning

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Coke remaining from the partial burning of coke deposited during the commercial re-forming of naphtha on a Pt-Re/Al₂O₃ catalyst was studied. Burning temperatures were 623–923 K, and the remaining coke was characterized by temperature-programmed oxidation, X-ray diffraction, electron diffraction, IR, ¹³C CP-MAS NMR, electron spectroscopy for chemical analysis, electron paramagnetic resonance, and chemical analysis. After coke is burned at 673 K, the residual coke shows the minimum value in the H/C ratio and the maximum in the thickness of the aromatic layers, degree of organization, C=O concentration, binding energy of C 1s, peak width, and *g* value. This agrees with the model of coke burning: at low temperatures, the burning is selective; the more hydrogenated and amorphous carbonaceous species are burnt first. At high temperatures, the burning is nonselective and all species are simultaneously burnt. Coke is partially oxidized during burning, and intermediate species with C=O and C—OH groups are formed.

Introduction

The carbonaceous deposit, or coke, formed on the naphtha re-forming catalyst during operation has a large molecular weight, containing polyaromatic hydrocarbons with paraffinic substitutions, and is poorly organized into pseudographitic structure (Biswas et al., 1987; Parera et al., 1987; Caruso et al., 1989). The degree of condensation of coke and its nature depend on the catalyst, type of feed, and operation parameters in the re-forming process (Beltramini et al., 1985; Barbier et al., 1985a,b; Franck and Martino, 1985; Parmaliana et al., 1987; Zhorov et al., 1980).

It has been shown that coke consists basically of two types of carbonaceous deposits: one of a low polymerization degree (H/C atomic ratio about 1) deposited on the metal, and the other of a higher degree of polymerization (H/C ratio about 0.5) deposited on the support (Parera et al., 1983; Barbier et al., 1980, 1985a,b). Coke is eliminated from the catalyst by burning with a diluted oxygen gas. The mechanism governing the burning process of the coke deposited in pelleted catalysts changes with temperature (Pieck et al., 1989). At high temperatures, the burning follows the shrinking core model; while at a low temperature, burning occurs according to the different reactivity of coke components and oxygen pressure.

In this paper, the coke remaining after partial burning at different temperatures was studied. The coke of commercially used catalyst pellets was partially burnt at various temperatures with analysis of the gaseous effluent during burning. After this partial burning, the catalyst pellets were finely ground and submitted to a temperature-programmed oxidation (TPO). After the coke was partially burned, the remaining coke was concentrated by acid elimination of the support and studied by X-ray diffraction (XRD) electron diffraction (ED), IR, ¹³C CP-MAS NMR, electron spectroscopy for chemical analysis (ESCA), electron paramagnetic resonance (EPR), and elemental C and H analysis.

Experimental Section

Catalyst. A Pt-Re-S/Al₂O₃ catalyst (1.5-mm-diameter and 5-mm-length extruded pellets) coked during a commercial re-forming operation was used. When the operation was started, the catalyst had a specific surface area

of 187 m² g⁻¹, a pore volume of 0.51 cm³ g⁻¹, and the following composition by weight: 0.3% Pt, 0.3% Re, 0.96% Cl, and 0.024% S. The catalyst was in operation for 6 months with a paraffinic naphtha at a pressure of 1.35 MPa and during this period accumulated 12.2% C in weight.

Coke Partial Burning. Coke partial burning was performed in equipment already described (Sad et al., 1980) that was modified to allow the entrance of the oxidant mixture. The procedure was as following: 4 g of the coked catalyst pellets were heated up to the burning temperature in a nitrogen flow, and then nitrogen was replaced by the oxidant mixture (1.9 vol % O₂ in N₂) during 2 h. The burning temperature varied between 623 and 823 K, the gas pressure was 0.1 MPa, and the gas flow rate was 125 mL(STP) min⁻¹.

Temperature-Programmed Oxidation. A Shimadzu Thermoanalyzer DT 30 was used, charging 6 mg of finely-ground catalyst. The heating rate was 20 K min⁻¹, and the flow rate of the previously dried oxidant mixture (1.9 vol % O₂ in N₂) was 50 mL min⁻¹. The same catalyst completely free of coke was used as reference.

Coke Concentration. For attainment of adequate signal levels, the concentration of coke was increased by dissolving alumina in an acid mixture, following a procedure proposed by other authors (Magnoux et al., 1987). A 1-g amount of the partially burnt catalyst ground to 25–80 mesh was attacked under vigorous stirring with a solution of 2 mL of 37 wt % hydrochloric acid and 8 mL of 40% hydrofluoric acid at room temperature for 6 h. After 1 night at room temperature, the suspension was filtered and the solid washed with distilled water, until the acids were eliminated, and dried at room temperature. The acid attack was repeated until no alumina was detected in the coke by XRD. Probably, this acid treatment causes no modification of the coke structure since coke in zeolites have been shown to be unaffected by zeolite dissolution in HF (Magnoux et al., 1987).

X-ray Diffraction. XRD spectra were obtained using a Rich Seifert ISO-Debyeflex 2002 powder diffractometer at 40 kV and 20 mA with nickel-filtered Cu K α radiation (wavelength = 1.54 Å). With the 2 θ values of the 002 plane peaks and the peak width measured at half the maximum height taken from the X-ray spectra, characteristic parameters of coke can be calculated.

Mean interlayer spacing of the aromatic system can be calculated with the Bragg equation:

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$$d = \frac{\lambda}{2 \sin \theta} \quad (1)$$

where λ (wavelength) = 1.54 Å.

Mean thickness of the aromatic layers can be calculated with the Scherrer equation (Anderson, 1985):

$$L = \frac{k\lambda}{Bj \cos \theta} \quad (2)$$

where k = Scherrer constant for which generally a value of 0.90 is taken for crystalline materials (Anderson, 1985) and Bj = peak width measured at half the maximum, in radians.

Infrared Spectroscopy. A Perkin-Elmer (Model 580B) double-beam spectrophotometer was used. The instrument range is 180–4000 cm^{-1} . Coke samples were pressed into disks with KBr.

Nuclear Magnetic Resonance. ^{13}C CP-MAS NMR spectra were recorded on a Bruker MSL-300 spectrometer equipped with a double bearing probe head and operated at 75.47 MHz. The spinning frequency was typically 3 kHz, and in order to prevent a superposition of spinning side bands and aliphatic peaks, the TOSS sequence—allowing a suppression of the former—was used. The contact time for cross polarization was 1 ms (the 90° value for ^1H is 6.2 μs), and the delay between each scan was 10 s. Depending on the amount of the product, 360–10000 scans were accumulated.

Electron Diffraction. Electron diffraction patterns were recorded with a JEOL 100 CS instrument. Samples were suspended in water and mounted on a 400-mesh copper grid with a nitrocellulose film.

Chemical Analysis by ESCA. XPS spectra were recorded with a Shimadzu ESCA-750 electron spectrometer using magnesium $K\alpha$ X-rays. The instrument working range is 0–1150-eV binding energy. Deconvolution of data was performed through a Shimadzu ESCAPAC 760 data system. For this analysis, samples were pressed at 0.35 MPa before the measurement.

Hydrogen-to-Carbon Ratio. Coke concentrated by the dissolution of alumina, using the acid treatment quoted above, was analyzed microgravimetrically for carbon and hydrogen.

In some cases, the H/C ratio of the burnt coke was obtained by chromatographic analysis of the combustion gases during the burning, using the method described elsewhere (Barbier et al., 1985a,b). From the coke combustion reaction, the H/C ratio can be deduced:

$$\frac{\text{H}}{\text{C}} = x = 4 \left(\frac{\text{O}_2 \text{ consumed}}{\text{CO}_2 \text{ produced}} - 1 \right) \quad (4)$$

In most cases, quantities of O_2 consumed and CO_2 produced are similar; then their ratio is near 1. Therefore, when eq 4 is used, great errors are possible. Another problem of this method is the consumption of oxygen without combustion when burning starts at a low temperature: when oxygen is fed, combustion starts slowly and a certain amount of oxygen is consumed by adsorption on the equipment and on coke. As little CO_2 is evolved, the calculation gives a higher H/C ratio than the correct value. At higher temperatures, the oxygen adsorbed or reacted with coke produces CO_2 without gaseous O_2 consumption, giving the calculation a lower H/C ratio than the correct value. For this reason, only the average value including all the combustion gases can be taken as a correct value.

Figure 1. (A) Temperature-programmed oxidation (TPO) of the original commercially coked catalyst. (B) TPO of a sample at which the TPO was previously performed up to 698 K. (C) TPO of a sample with previous TPO up to 773 K.

Electron Paramagnetic Resonance (EPR). A Bruker Model ER-200 spectrometer operating in band X (9.7 GHz) at room temperature was used.

Results and Discussion

Figure 1 shows the TPO of the original coked catalyst, A, and of samples B and C obtained when previous TPO runs were stopped at 698 and 773 K, respectively. The original coked catalyst presents two burning zones. The first zone—at 573–723 K—corresponds to the burning of the coke over the metallic function (Pt-Re). The second zone corresponds to the burning of the coke over the acid function ($\text{Al}_2\text{O}_3\text{-Cl}$) that is more polymerized and poorer in hydrogen (Parera et al., 1983; Barbier et al., 1980, 1985a,b). In sample B, all the coke on the metal and a very small fraction on the support were eliminated. In sample C, the burning of coke over the support continued. This shows that the TPO is selective; the coke on the metal is burnt first, and the burning continues with coke increasingly difficult to burn each time (more polymerized) and located on the support. TPO is performed with a fine powder and there is no mass-transfer limitation, at least when the temperature is not very high.

In the commercial burning of the coked catalyst pellets, the process starts at a low temperature and with a very low oxygen concentration; consequently more hydrogenated coke (located on the metal) is burned. When this burning is already finished, temperature and oxygen concentration increase slowly, thus allowing the burning of more coke. The burning starts selectively, controlled by the chemical step, but when the temperature is high, the controlling step can change. The oxygen diffusion can become the limiting step, and the burning will be nonselective, with all the coke of external shells of the catalyst being eliminated first. When the experiment is performed isothermally, as in our case, a selective burning can be obtained at a low temperature and a nonselective one at a high temperature.

Table I shows the carbon remaining on the catalyst after burning during 2 h at various temperatures, the H/C atomic ratio of the residual coke, the interlayer spacing of the aromatic layers, and the mean thickness of these layers obtained by XRD in the coke concentrated after elimination of alumina by acid treatments.

During the 2-h burning, the catalyst bed temperature remained constant because of the great thermal capacity

Table I. Characteristics of Coke Remaining over the Partially Burnt Coked Catalysts

burning temp, K	residual carbon, %	atomic ratio (H/C)	interlayer spacing, Å	mean thickness, Å
sample without burning	12.2	0.56	3.41	25.6
623	10.0	0.52	3.40	28.9
673	8.5	0.40	3.42	29.1
723	7.3	0.48	3.42	30.2
773	6.8	0.56	3.42	28.1
823	5.2	0.57	3.43	21.4

Figure 2. Electron-diffraction pattern of coke from the sample without burning treatment.

of the system: the catalyst bed has a small ring-shaped cross section (between thermocouple well and reactor wall), the reactor wall is 2 cm thick, and the gas has 98.2% N_2 . The measured burning temperature is the average particle-gas temperature, but the actual temperature inside the catalyst particle could be higher. The rate of burning is rather small because in all cases a great amount of carbon remains after the 2-h burning.

The original coke has an H/C ratio of 0.56, and the residual coke, when the partial burning is performed at 673 K, has a ratio of 0.4. At higher temperatures, the residual coke has the same H/C ratio as that of the original coke. This can be explained considering that coke burning is selective at low temperatures (up to 723 K), burning first species richer in hydrogen, leaving on the catalyst a heavier coke (lower H/C ratio). Sotirchos et al. (1983) found that hydrocarbonaceous species are five times more reactive to oxygen than the graphitic ones. At high burning temperatures (773–823 K), all coke components are simultaneously burnt, and the residual coke has a H/C ratio similar to values of the original coke. This agrees with the results of Pieck et al. (1989), who stated that, at low temperatures, the burning is nearly uniform in all the catalyst particles (controlled by the chemical step) and, at high temperatures, the burning follows the shrinking core model (controlled by the oxygen diffusion step).

It can be seen (Table I) that the mean interlayer spacing is practically independent of the burning temperature and larger than the one of graphite ($d_{002} = 3.354$ Å). The interlayer spacing is larger than that in graphite because coke is a disordered carbonaceous material formed by polymeric aggregates with alkyl chains, as quoted before (Bakulin et al., 1974; Parera et al., 1984). The mean thickness of the aromatic layers as a function of burning temperature has a maximum.

Figures 2 and 3 show the electron diffraction patterns of the sample without burning treatment and of the sample partially burnt at 673 K, respectively. In a comparison of

Figure 3. Electron-diffraction pattern of coke from the sample partially burnt at 673 K.

Figure 4. Infrared absorption spectra of coke from the original samples, without burning treatment, and from samples partially burnt at the indicated temperature.

all diagrams, the sample without treatment shows an amorphous structure, while the residual coke of the burning at low temperatures presents ordered structures more evident in the sample burnt at 673 K. This means that, at a low temperatures, the smaller and less ordered coke particles burn first. At a high burning temperature and because of the shrinking core mode of burning, the residual coke has a structure similar to that of the original material before treatment.

Figure 4 shows IR (1000–1800- cm^{-1}) spectra of the original coke and of that from the partially burnt samples. Using the equipment computer, it was given the same intensity to the peak at 1680–1480 cm^{-1} in all the spectra in order to take it as a reference to compensate differences in coke concentration and thickness of the disks. The absorption band appearing between 1480 and 1680 cm^{-1} in all samples can be ascribed to the stretching of the C=C bond belonging to olefins, aromatic rings, and polyaromatics. The peak at about 1400 cm^{-1} can be ascribed to the CH bond stretching in C-(CH₃)₂ or C-(CH₃)₃ groups. The absorption peak at about 1200 cm^{-1} is due to the stretching of the CH group in the aromatic plane (Rouxhet et al., 1980).

In a comparison of the absorption spectra of all samples, it can be seen that there is no absorption due to C-OH (3400–3600 cm^{-1} , not shown in Figure 4, and 1100 cm^{-1}) and to C=O (1710 cm^{-1}) in the sample without burning treatment. Meanwhile, samples partially burnt present

Table II. Binding Energy of C 1s, Peak Width, and *g* Values of Coke from the Partially Burnt Coked Catalysts

burning temp, K	binding energy, eV	full width at half-maximum, eV	<i>g</i>
sample without burning	286.9	3.6	2.0025
623	286.8	3.3	2.0027
673	287.2	4.0	2.0031
723	287.2	3.0	2.0025
773	286.8	2.7	2.0025
823	286.6	2.5	2.0024

an absorption peak approximately at 1715 cm⁻¹ due to the group C=O and another peak at 1100 cm⁻¹. This last one can be due to the presence of OH, because a broad peak in the range 3400–3600 cm⁻¹ also appears. It can be seen that the intensity of the peak produced by C=O has a maximum value at 673 K, which corresponds to an intermediate coke burning. Then, coke burning follows the following consecutive reaction

which is similar to the model proposed by Furimsky et al. (1988) for coke deposited on nickel molybdate hydro-treating catalyst.

The peak in the range 1480–1680 cm⁻¹ moves to a higher frequency from the original sample to the one burnt at 673 K, moving then to lower frequencies at high burning temperatures (723–823 K). This could indicate that the structure of residual coke should be more organized in the sample burnt at 673 K because of its highest vibration energy, being in agreement with the results of transmission electron microscopy (TEM) analysis above.

Solid-state NMR spectra show only three broad absorptions around 125, 73, and 25 ppm attributed to olefinic and aromatic carbons (125 ppm), carbon singly bonded to oxygen (75 ppm), and -CH₂CH₃ peaks (25 ppm). The main difference between spectra of coke samples is the relative amount of oxygen bonded to carbon: this peak can be very easily observed in samples burned at 623 and 673 K. In the same range of temperatures, IR data show the greatest absorption peak due to the C=O group, the position of the maximum not being the same. In NMR, the maximum is in the sample burnt at 623 K, while in IR spectra, the C=O group at 1715 cm⁻¹ shows a maximum in the sample burnt at 673 K. This may be due to a different structure of the oxidized species observed in each case. If there is less hydrogen in the neighborhood of the C=O group, the intensity measured by CP-MAS will be lower. No carbon in the C=O group was detected in NMR, because there is no hydrogen directly bonded to the carbon, and so cross polarization cannot be efficient in this case. The maximum in C=O cannot be detected in NMR.

C 1s and O 1s spectra of the samples were recorded; the C 1s peak of the sample was near that of the high vacuum pump oil and was resolved by deconvolution. The O 1s peak is due to oxygen chemically bonded to the sample (C=O and C-OH). Table II shows the values of binding energy and peak width of C 1s, and the values of factor *g* obtained by EPR. It can be seen that the peak width, its position, and factor *g* have a maximum value. According to Wagner et al. (1978), the shift toward higher values of binding energy of C 1s is probably due to the formation of carbon-oxygen species in which the C 1s peak has higher values of binding energy. The peak broadening can be due to the higher heterogeneity of the samples due to the simultaneous presence of oxidized and nonoxidized coke. The sample burnt at 673 K was the only one in which oxygen of oxidized coke was found. The calculated O/C atomic ratio was 0.02. In the other samples, the

Figure 5. H/C atomic ratio of the fraction of coke elimination as a function of the percentage of this coke burnt at (●) 698 and (■) 823 K.

oxygen surface concentration was below the detection limit of the equipment.

Changes in binding energy and peak width of C 1s (and also the presence of O 1s in the sample regenerated at 673 K) with the burning temperature agree with IR results (presence of C=O and C-OH passing through a maximum as a function of burning temperature) and the other techniques (TEM, XRD). Therefore, at a low oxidation temperature, an oxidized coke is produced; at a high temperature, the period of life of C=O species is reduced, and the burning rapidly produces CO₂ and H₂O.

Taking into account the peak appearing around 1715 cm⁻¹ in the IR spectrum (C=O groups) and the aromatic ratio O/C of the sample burnt at 673 K in the ESCA spectrum, it is possible to calculate oxygen surface concentration for each sample. The values in weight percent are 1.6, 2.6, 1.3, 0.5, and 0.2 for samples partially burnt at 623, 673, 723, 773, and 823 K, respectively.

From ESR data, the sample burnt at 673 K has the highest value of *g*, probably due to the presence of oxygenated groups in the proximity of aromatic groups (Marchand and Canard, 1980).

In Figure 5, the H/C atomic ratio of the fraction of coke eliminated is plotted as a function of the percentage of this coke. The combustion was performed at 0.34 MPa with a flow rate of 42 mL min⁻¹ of oxidative mixture containing 5 vol % O₂ in N₂ and at two temperatures, 698 and 823 K. To calculate the H/C ratio, average concentrations of CO₂ and O₂ were chromatographically determined in the period at which the coke was eliminated. At 698 K, the H/C value decreases when the amount of coke eliminated increases, but it remains constant at 823 K. This agrees with concepts already quoted: at a low temperature, the burning is selective, the more hydrogenated coke being eliminated first, while, at a high temperature, the burning is nonselective following the shrinking core model.

Conclusions

Different techniques agree regarding the phenomenon of burning and the characteristics of coke during its burning. At low temperatures, the burning is selective, the more hydrogenated fraction with smaller aggregate sizes being first eliminated, and surface C=O and C-OH groups are formed as intermediates. At higher temperatures, these groups rapidly complete the oxidation to CO₂, and the burning is nonselective: all coke components are burnt from the external layers to the inner part (shrinking core model).

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