

MODELLING OF REGENERATION PROCESS OF ZEOLITE-CONTAINING CRACKING CATALYSTS

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Abstract

The research aimed at discovering the patterns of the zeolite-containing cracking catalysts regeneration, which are required for the mathematical model development, is presented. This model takes into account the physicochemical and kinetic patterns of the of coke oxidation reactions in the air. The numerical calculations using the model allowed to predict of the regeneration temperature, the coke amount on the regenerated catalyst and the combustion gas composition depending on the process conditions of the reactor-regenerator unit and the coke amount on the dead catalyst.

Keywords: catalytic cracking; zeolite-containing catalyst; coke, regeneration; mathematical modeling.

1. Introduction

The catalytic cracking is the most important producer of the high-octane gasoline components and feedstock for the petrochemical industry all over the world and contributes to the oil refining deepening. The catalytic cracking capacity exceeds 35% of the oil refining volume in the US, 15% in Western Europe and less than 10% in Russia. Currently, the total global capacity of the catalytic cracking is about 700 million tons of feedstock per year [1]. The catalytic cracking efficiency is determined by the activity, the selectivity and the stability of the microspheric zeolite-containing catalysts primarily. However, the deterioration of these indicators during the operation is observed due to the catalysts deactivation by coke and heavy metals, abrasion, sintering, etc.

The catalysts deactivation by coke is a reversible process. In industrial conditions, the catalyst undergoes regeneration in order to restore the activity. During this process, the coke oxidation using the oxygen-containing mixtures and partial reduction of the catalytic properties occur, and the catalysts operation becomes cyclical as "operation-regeneration" process [2-3].

The oxidation rate of a coke can be intensified by increasing the oxygen amount in the supplied air to regeneration, and by introducing the oxidation promoters into the catalysts, which do not have a noticeable effect on the activity and selectivity of the catalysts.

At high oxidation rates, a sharp increase in the regeneration zone temperature occurs. This fact can become the main reason for the irreversible catalyst deactivation as a result of sintering the pore structure [4-7]. The problem of deactivation due to overheating is so the most important for regenerated catalysts, and it is required to reduce the oxidation rates instead of intensifying the coke oxidation.

Also, the heat released during the coke oxidation is used to conduct the process in the reactor. Heat transfer is carried out by heating the catalyst flow in the regenerator.

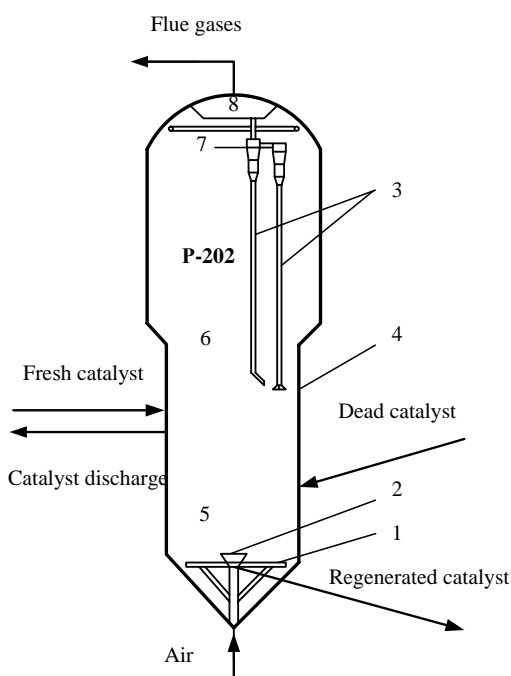
Thus, the oxidative regeneration efficiency has a significant effect on the economics of the catalytic processes, and it determines the competitiveness of various design options for catalytic cracking.

The development and application of mathematical model taking into account the physicochemical and kinetic patterns of the coke oxidation reactions with the air [8-17] allow to predict not only the residual coke amount on the catalyst and the composition of the flue gases of regeneration, but also the regeneration zone temperature depending on the process conditions of the reactor-regenerator block taking into account the amount of coke formed on the catalyst surface in the reactor.

The research aims to develop the mathematical model of the regeneration of zeolite-containing catalysts in catalytic cracking to forecast the oxidized coke amount on the catalyst, the regeneration temperature, and the flue gas composition.

2. Object and methods of research

The object of research is the oxidative regeneration process of the catalytic cracking unit (section S-200), which is a part of KT-1/1 combined unit for deep processing of fuel oil. A microspheric zeolite-containing catalyst with an average particle size of 90 μm bulk and an apparent density of 840 and 1400 kg/m^3 is circulated between the reactor and the regenerator at the catalytic cracking unit.



Along with the target and by-products (unstable gasoline, rich gas, 195-510°C fraction, 310-420°C fraction, 420+°C fraction), the coke deposition on the zeolite-containing catalyst surface occurs during operation. The oxidative regeneration in the fluidized catalyst bed is carried out in order to partially catalyst reduction on the KT-1/1 unit. The regenerator capacity for oxidized coke is 13600 kg/h. The regenerator is a cylindrical apparatus with internal diameters of 9 and 11 m and a height of 27.6 m. The effective regenerator volume is 1800 m^3 with an average height of the fluidized catalyst bed of 4 m (Fig. 1).

Fig. 1. The regenerator scheme of the KT-1/1 unit: 1-boxes and manifolds; 2-funnel; 3-2-stage cyclones; 4- regenerator shell; 5 - fluidized bed of the catalyst; 6-zone of the dilute-phase fluidized bed of the catalyst; 7- sedimentation zone; 8 - collecting chamber

After the oxidative regeneration stage, the regenerated catalyst from the regenerator P-202 is gravity feed to the reactor for the compounding with the feedstock. The main process conditions of the reactor-regenerator block are given below (Tab.1). The material balance of the KT-1/1 regenerator is given below (Tab.2).

The following methods were used during the modeling the oxidative regeneration of cracking catalysts and the studying the characteristics of cracking catalysts at the "operation-regeneration" cycle:

1. Granulometric analysis for determining the particle diameter distribution at catalyst sample using HORIBA LA-300 laser meter (detection limit 0.1 to 600 μm in diameter using a laser diode, a maximum error of 0.6%).
2. A mathematical modeling method for the mathematical description of the regeneration of zeolite-containing cracking catalysts in order to forecast the residual coke on the regenerated catalyst, the regeneration temperature, and the flue gas composition.

Table 1. Operating factors and process conditions of the reactor-regenerator block of the KT-1/1 unit

Operating factors and process conditions	Value
Feedstock flowrate to reactor, tons per day	6686.2
Catalyst circulation ratio, $\text{ton}_{\text{cat}}/\text{ton}_{\text{feed}}$	7.95
Mass of the catalyst in the regenerator, ton	150–200
Coke amount on the coked catalyst, % wt	0.47
Residual coke amount on the catalyst, % wt	0.045
Catalyst flow temperature at the regenerator inlet, K	808.7
Air consumption in regenerator, m^3/h at standard conditions.	167 522.2
Air temperature at the regenerator inlet, K	410.6
Regeneration zone temperature, K	903–1003
Air pressure at the regenerator inlet, MPa	0.42
Pressure in the regenerator sedimentation zone, MPa	0.17
Amount in flue gases of regeneration, % vol:	
– CO/CO ₂	0.0009/11.86
– O ₂ /N ₂	4.94/83.19

Table 2. The material balance of the KT-1/1 regenerator (calculation)

Flow	Value tons per day	%
Input:		
– catalyst	53 219.8	95.84
– coke after reactor	251.3	0.45
– air	2 060.9	3.71
total	55 532.0	100.00
Output:		
– catalyst	53 219.8	95.83
– coke after regenerator	54.3	0.098
– flue gases	2 236.7	4.03
total	55 510.8	99.96

3. Development of regeneration model of zeolite-containing cracking catalysts

The main stages for the modeling the oxidative regeneration of zeolite-containing cracking catalysts are the following:

- conducting the thermodynamic analysis of all possible reactions in the regenerator;
- determination of the hydrodynamic mode of the regenerator in the fluidized bed of the catalyst;
- establishing the probable scheme for coke oxidation on the surface of the cracking catalyst;
- development of a kinetic and mathematical model of the regeneration;
- verification of the mathematical model.

3.1. Thermodynamic analysis of the oxidative regeneration reactions

A calculation of the thermodynamic functions changing of the model reactions at the temperature range from 808 to 1003 K and a pressure of 1 bar was made in order to estimate and forecast the reactions probability and the carbon transformations products formation [18]. This temperature range corresponds to the regenerator process conditions. As it is known, the coke compounds on the surface of the catalyst are characterized by a complicated structure, and the thermochemical information for such compounds is limited in the literature. Therefore, it was accepted to investigate the coke structures reactions on the example of the combustible component – carbon in the oxygen atmosphere of air and water vapor (Tab.3).

Table 3. The dependence of the thermodynamic functions changing of model reactions at a temperature $T = 808 \div 1003$ K and pressure 1 bar

Reaction	kJ/(mol·K)		kJ/mol	
	$\Delta_r S_T^0$	$\Delta_r H_T^0$	$\Delta_r G_T^0$	
$2C_{\text{coke}} + O_2 = 2CO$	$0.169 \div 0.166$	$-(226.35 \div 229.10)$	$-(362.97 \div 395.19)$	
$2CO + O_2 = 2CO_2$	$-(0.167 \div 0.165)$	$-(562.76 \div 561.20)$	$-(427.60 \div 395.36)$	
$C_{\text{coke}} + O_2 = CO_2$	$0.001 \div 0.000$	$-(394.56 \div 395.15)$	$-(395.28 \div 395.27)$	
$C_{\text{coke}} + H_2O_{\text{vapor}} = CO + H_2$	$0.127 \div 0.124$	$127.81 \div 125.71$	$25.00 \div 0.90$	
$C_{\text{coke}} + CO_2 = 2CO$	$0.168 \div 0.165$	$168.20 \div 166.05$	$32.31 \div 0.09$	

The thermodynamic analysis results of model reactions showed that the exothermic reactions (1) – (3) ($\Delta_r G_T^0 < 0$) predominate and the endothermic reactions (4)–(5) are improbable ($\Delta_r G_T^0 > 0$) under regenerator process.

3.2. Determination of the hydrodynamic mode of the regenerator

Hydrodynamic calculations [19-20] showed that the catalyst bed in the regenerator passes into the fluidization state at the flue gas velocity of 0.0014 m/s (calculated on the free area by (1)):

$$\omega_{if} = 0,005 \cdot \varepsilon^3 \cdot \frac{d^2 \cdot (\gamma_k - \gamma_a)}{\mu \cdot (1 - \varepsilon)}, \quad (1)$$

$$\omega_{sv} = \sqrt{\frac{4}{3} \cdot \frac{g \cdot d \cdot (\gamma_k - \gamma_a)}{C \cdot \gamma_a}}, \quad (2)$$

The destruction of the fluidized catalyst bed, which is characterized by the elutriation of the solid particles from a fluidized bed, occurs when the velocity is above than the suspension velocity of 0.23 m/s (2):

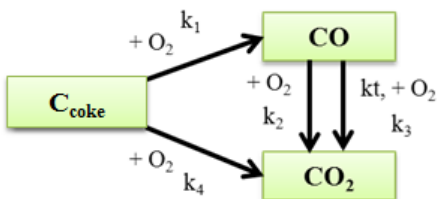
The actual velocity of the regeneration flue gas (3) is 0.69 m/s (3):

$$\omega_{av} = \frac{CF \cdot Q_t \cdot P_s \cdot (T_b + T_f)}{(P_b + P_f) \cdot T_b} \cdot 3600 \cdot A_t \quad (3)$$

here d – the average (effective) diameter of the catalyst particle, m; γ_k – the apparent density of the catalyst, kg/m^3 ; γ_a and μ – the density (kg/m^3) and the dynamic viscosity (kgf/m^2) of the fluidizing agent; g – the intensity of gravity, m/s^2 ; c – the resistance coefficient; CF – the coke accounting constant in the regenerator (1,035); Q_t – the total air flow to regenerator, m^3/h at standard conditions; P_s – the atmospheric pressure, kgf/cm^2 ; T_b – the standard temperature (273.15) K; T_f – the temperature in the regeneration zone, K; P_b – barometric pressure, kgf/cm^2 ; P_f – the average absolute pressure in the catalyst bed, kgf/cm^2 ; A_t – sectional area of the regenerator (89.88 m^2).

It can be considered that the fluidized bed is an approximation to the plug-flow reactor because the actual velocity of regeneration flue gases exceeds the value of the suspension velocity ($\omega_{if} \ll \omega_{sv} \ll \omega_{av}$).

3.3. The development of the kinetic model of the oxidative regeneration of zeolite-containing cracking catalysts



The results of the thermodynamic analysis of all possible reactions in the regenerator at the temperature range 808- 1003 K allow suggesting a conversion scheme during the regeneration (Fig. 2).

Fig. 2. The scheme of coke oxidation on the catalytic cracking catalyst

By the coke oxidation scheme on the cracking catalysts surface, the mathematical model of the oxidative regeneration in the fluidized catalyst bed in steady state conditions of the plug-flow reactor was developed (4) (the assumption of quasi-homogeneity of the components was adopted):

$$\left\{ \begin{aligned} \frac{dC_{C_{\text{coke}}}}{d\tau} &= -2 \cdot k_1 \cdot \left(\frac{\rho_{fb} \cdot X_{\text{coke}}^{\text{deadcat}}}{MM_{\text{coke}}} \right)^2 \cdot c_{O_2} - k_4 \cdot \left(\frac{\rho_{fb} \cdot X_{\text{coke}}^{\text{deadcat}}}{MM_{\text{coke}}} \right)^2 \cdot c_{O_2}; \\ \frac{dC_{O_2}}{d\tau} &= -k_1 \cdot \left(\frac{\rho_{fb} \cdot X_{\text{coke}}^{\text{deadcat}}}{MM_{\text{coke}}} \right)^2 \cdot c_{O_2} - k_2 \cdot c_{CO}^2 \cdot c_{O_2} - k_3 \cdot c_{CO}^2 \cdot c_{O_2} - k_4 \cdot \left(\frac{\rho_{fb} \cdot X_{\text{coke}}^{\text{deadcat}}}{MM_{\text{coke}}} \right)^2 \cdot c_{O_2}; \\ \frac{dC_{CO}}{d\tau} &= 2 \cdot k_1 \cdot \left(\frac{\rho_{fb} \cdot X_{\text{coke}}^{\text{deadcat}}}{MM_{\text{coke}}} \right)^2 \cdot c_{O_2} - 2 \cdot k_2 \cdot c_{CO}^2 \cdot c_{O_2} - 2 \cdot k_3 \cdot c_{CO}^2 \cdot c_{O_2}; \\ \frac{dC_{CO_2}}{d\tau} &= 2 \cdot k_2 \cdot c_{CO}^2 \cdot c_{O_2} + 2 \cdot k_3 \cdot c_{CO}^2 \cdot c_{O_2} + k_4 \cdot \left(\frac{\rho_{fb} \cdot X_{\text{coke}}^{\text{deadcat}}}{MM_{\text{coke}}} \right)^2 \cdot c_{O_2}; \\ \frac{dT}{d\tau} &= \frac{k_1 \cdot \left(\frac{\rho_{fb} \cdot X_{\text{coke}}^{\text{deadcat}}}{MM_{\text{coke}}} \right)^2 \cdot c_{O_2} \cdot \Delta H_1 + k_2 \cdot c_{CO}^2 \cdot c_{O_2} \cdot \Delta H_2 + k_3 \cdot c_{CO}^2 \cdot c_{O_2} \cdot \Delta H_3 + k_4 \cdot \left(\frac{\rho_{fb} \cdot X_{\text{coke}}^{\text{deadcat}}}{MM_{\text{coke}}} \right)^2 \cdot c_{O_2} \cdot \Delta H_4}{\rho^f \cdot c_p^f} \end{aligned} \right. \quad (4)$$

here $dC_i/d\tau$ – change in the concentration of the i-th reaction reagent by contact time, mol/(m³·s); k_i – the rate constant of the i-th reaction according to the Arrhenius equation, c⁻¹; ρ_{fb} – the density of the fluidized bed of the catalyst, kg/m³; $X_{\text{coke}}^{\text{deadcat}}$ – coke quantity on the coked catalyst; $X_{\text{coke}}^{\text{deadcat}} = 0 \dots 1$; MM_{coke} – the molecular weight of coke, kg/mol; c_i – the current concentration of the i-th reaction reagent, mol/m³; ρ^f – the density of the reaction mixture, kg/m³; c_p^f – the specific heat of the reaction mixture, kJ/(kg·K); $dT/d\tau$ – the temperature change in the regenerator by contact time, K/s; ΔH_j – the thermal effect of the corresponding reactions, kJ/mol; the initial conditions: $\tau=0$; $C_{\text{coke}}=C_{\text{coke},0}$; $C_{O_2}=C_{O_2,0}$; $C_{CO}=0$; $C_{CO_2}=0$; $T=T_0$.

The contact time in the volume of the is calculated according to (5):

$$\tau = \frac{V_{fb}}{V_{\text{coke}} + V_{\text{air}}}, \quad (5)$$

here V_{fb} – the volume of the fluidized bed of the catalyst, m³; V_{coke} – the coke amount on the catalyst surface from the reactor, m³/s; V_{air} – the total air flow for regeneration, m³/s.

The software implementation of the model is carried out using the PascalABC.NET integrated programming software.

The kinetic parameters of the oxidative regeneration reactions of the zeolite-containing cracking catalysts presented below were determined by solving the inverse kinetic problem using the experimental data from the industrial catalytic cracking unit for a long period of its operation. The activation energy values of the reactions are taken according to the literature data review [21].

Also, at the stage of the kinetic parameters establishing, it is important to estimate the pore-diffusion resistances presence for determining the kinetic region boundaries. Also, in accordance with [22], the regeneration of the microspherical cracking catalysts in the fluidized bed is characterized by the kinetic region at temperatures up to 923 K, by the transition region at 923–943K and by the diffusion region at temperatures above 943 K.

Estimation of the pore-diffusion resistances presence by the Thiele modulus (6) and the effectiveness factor (7) values for an average particle size of 90 μm showed that the pore-

diffusion resistances of the regeneration reactions on the zeolite-containing cracking catalysts are absent.

$$\psi = L \sqrt{\frac{kC_0^{n-1}}{D_{eff}}} \tag{6}$$

$$\eta = \frac{3}{\psi} \cdot \left(\frac{e^\psi + e^{-\psi}}{e^\psi - e^{-\psi}} - \frac{1}{\psi} \right), \tag{7}$$

here L – the catalyst grain size equal to the volume to the outer surface ratio, m , k – the reaction rate constant, n – the reaction order, C_0 – the reagent concentration, mol/l, D_{eff} – effective diffusion factor, m^2/s .

It is connected with the Thiele modulus value does not exceed $\psi \leq 1$ for first-order reactions, and the effectiveness factor of the catalyst grain use is ~ 1 (Tab.4).

The developed model of the regenerator allows forecasting the coke amount on the regenerated catalyst, the flue gases composition, the regeneration temperature.

Table 4. The kinetic parameters of regeneration at $T = 945$ K and $P = 0.17$ MPa

Reaction	k_0, s^{-1}	$E_a, kJ/mol$	$k(T), s^{-1}$	Thiele modulus	Effectiveness factor
$2C_{coke} + O_2 = 2CO$	$7.92 \cdot 10^5$	99.1	2.62	0.10	0.999
$2CO + O_2 = 2CO_2$	$6.52 \cdot 10^3$	50.4	10.65	0.21	0.997
$2CO + O_2 = 2CO_2$ (catalytic)	$8.38 \cdot 10^5$	90.8	7.98	0.18	0.998
$C_{coke} + O_2 = CO_2$	$2.90 \cdot 10^5$	65.2	71.90	0.54	0.981

3.4. Verification of the mathematical model

Calculated and experimental data comparison from the industrial catalytic cracking unit showed that the mathematical model describes the process with high accuracy (Fig. 3, 4), this model takes into account the physico-chemical and kinetic patterns of the coke oxidation reaction with the air.

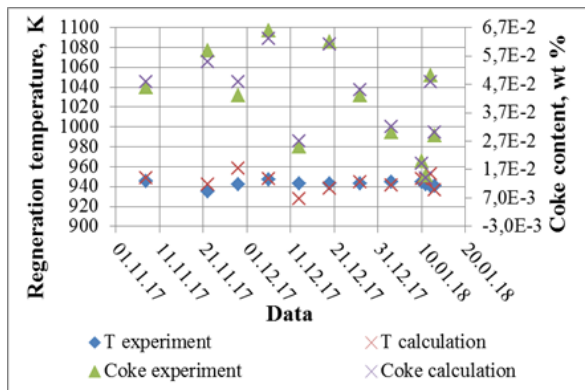


Fig. 3. Comparison of calculated and experimental data in the residual coke amount and the regeneration temperature

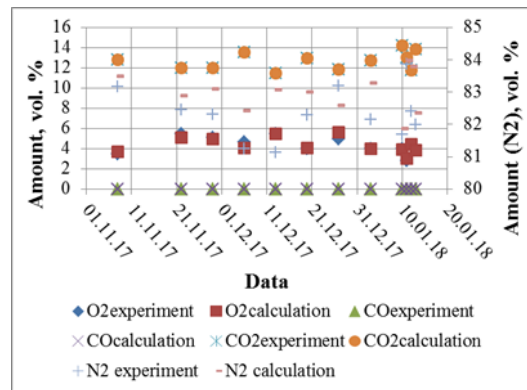


Fig. 4. Comparison of calculated and experimental data in the flue gases of regeneration (O_2 , CO , CO_2 , N_2)

The average relative errors are 5.6% at the calculation of the coke amount on the regenerated catalyst, 5.3, 4.8%, 4.9 %, 0.9% at calculations of O_2 , CO , CO_2 , the N_2 amount in the flue gas of regeneration relatively.

In addition, a different dynamics of coke oxidation is observed depending on the various initial conditions (temperature and air consumption, initial coke concentration and catalyst

temperature from the reactor outlet). The use of the developed model will ensure accounting of these factors at optimization of the reactor and regenerator process conditions.

4. Conclusions

The thermodynamic and kinetic patterns of the oxidative regeneration of zeolite-containing cracking catalysts were discovered by the proposed coke oxidation scheme. It was determined that the coke oxidation reactions with the carbon dioxide formation ($\Delta G_{av} = -395.28$ kJ/mol, $k = 71.90$ s⁻¹) and the afterburning of carbon monoxide in the gas phase ($\Delta G_{av} = -411.48$ kJ/mol, $k = 10.65$ s⁻¹) are characterized by the greatest thermodynamic probability and the highest reaction rates. Also, the least intensive reaction under process conditions is the coke oxidation reaction with carbon monoxide formation ($\Delta G_{av} = -395.19$ kJ/mol, $k = 2.62$ s⁻¹).

The adequate mathematical model of the oxidative regeneration of zeolite-containing cracking catalysts with justification the plug-flow reactor mode was developed (the maximum relative error is 5.6%).

The mathematical model allows forecasting the coke amount on the regenerated catalyst, the flue gases composition and the regeneration temperature depending on the process conditions of the catalytic cracking reactor and regenerator.

The application of the mathematical model allows to optimize the reactor and the regenerator process conditions, to prevent the irreversible deactivation of the catalyst (thermal deactivation), to forecast the residual coke and the catalyst activity, and to estimate the amorphism degree of coke regarding C/H ratio.

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