



SELECTION OF KEY PARAMETERS FOR GREEN COKE CALCINATION IN A TUBULAR ROTARY KILN TO PRODUCE ANODE PETCOKE

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ABSTRACT

The article deals with the main problems of a modern industrial technology for producing calcinated petroleum coke from green coke in a tubular rotary kiln. A high quality calcinated coke is obviously necessary both for metallurgy and chemical industry, hence its properties should meet the corresponding high requirements. Petroleum coke is a nice alternative to anthracite coke. Since the total volume of oil refining in the world is high, petcoke production has a stable raw material base, unlike anthracite, which deposits are being gradually depleted. Physical and chemical properties of petcoke and coal coke are similar in many aspects, but a proper anthracite coke sometimes demonstrates better and more stable characteristics compared to ordinary petroleum coke. Moderate or low quality of a definite batch of petroleum coke often limits its possible areas of application. The reason is usually a non-optimal calcination procedure that results in improper internal structure and compositional non-homogeneity of a batch of commercial petroleum coke. Improving the situation is necessary for many reasons. Obtaining the high-quality coke for electrodes production is important for aluminium industry. Calcination is the main technological stage where composition homogeneity and structure peculiarities of a definite coke are formed. To meet the technology requirements one should determine the key parameters of calcination process and establish a proper relationship between the regulated parameters and the product quality indicators.

Keywords: petroleum coke, calcined coke, anodic coke, calcination, calcining control, calcination temperature, rotary kiln.

INTRODUCTION

Crude oil is a complicated mixture of hydrocarbons of various molecular mass and structure. Modern oil refining technologies are generally focused at maximum production of light and medium fractions from raw oil. With the deepening oil refinery the amount of light hydrogen-containing components in the processing oil gradually decreases from stage to stage until a wax or semi-solid oil residue remains. This residue is one of raw materials for green petroleum coke production [1, 2]. Highly aromatic residues from the basic secondary processes of oil refinery - catalytic cracking, pyrolysis - give other valuable components for petroleum coke production.

Raw (or green) coke after a semi-batch coking column is a semi-solid, relatively inert substance, consisting mostly of elementary carbon. The approximate composition of raw coke is presented in Table-1 [3, 4, 5].

Table-1. Elementary petcoke composition.

Component:	Ratio, %
C	91,0 - 99,5
H	0,035 – 4,0
S	0,5 – 8,0
(N+O)	1,3 – 3,8
Metals	Residue

Regarding to physical, chemical properties and structure of the granules, petroleum coke is close to coal (anthracite). Sometimes coal and petroleum cokes are used

together or as an alternative to each other. Actual rates of oil extraction and its further refining also stimulate the production of petroleum coke. The production of petroleum coke has nowadays become cheaper than coal mining and coal coke production. Beyond that, the world's resources of high-quality anthracite coal are rapidly decreasing, while oil refinery plants have got a stable raw material base for petcoke production in necessary amount. The range of a definite coke application is totally determined by its quality. The quality of petroleum coke follows from the degree of its purity and physical and chemical properties. Green coke granules are usually formed by unstructured crystallites connected with valence bonds that are not strong enough. To become a commercial product it should be calcinated. The internal structure and composition of petcoke grains directly define the main product properties [4, 6, 7].

High electrical conductivity is obligatory for electrode coke to be used in non-ferrous metallurgy, especially in the aluminum production industry. Green coke initially has a rather high internal electric resistance. Low electrical conductivity is invaluable for petcoke potential applications [8].

MATERIALS AND METHODS

The main methods in this study were the analysis of literature data and mathematical 3D modeling of the calcination process in the volume of Petcox particles.

Grains of pure carbon with a fiber texture perfectly conduct electric current and heat. Green petcoke should be calcined to provide an optimal structure. Usually coke calcination process is carried out in tubular rotary kilns (see Figure-1). High-temperature processing helps to clean the coke from impurities and to form an optimal



internal structure of its grains. In case that calcinating procedure is run correctly, the calcined coke should acquire new properties, including high thermal and

electrical conductivity, heat resistance and mechanical strength. Major part of such coke is used for the production of electrode materials [9].

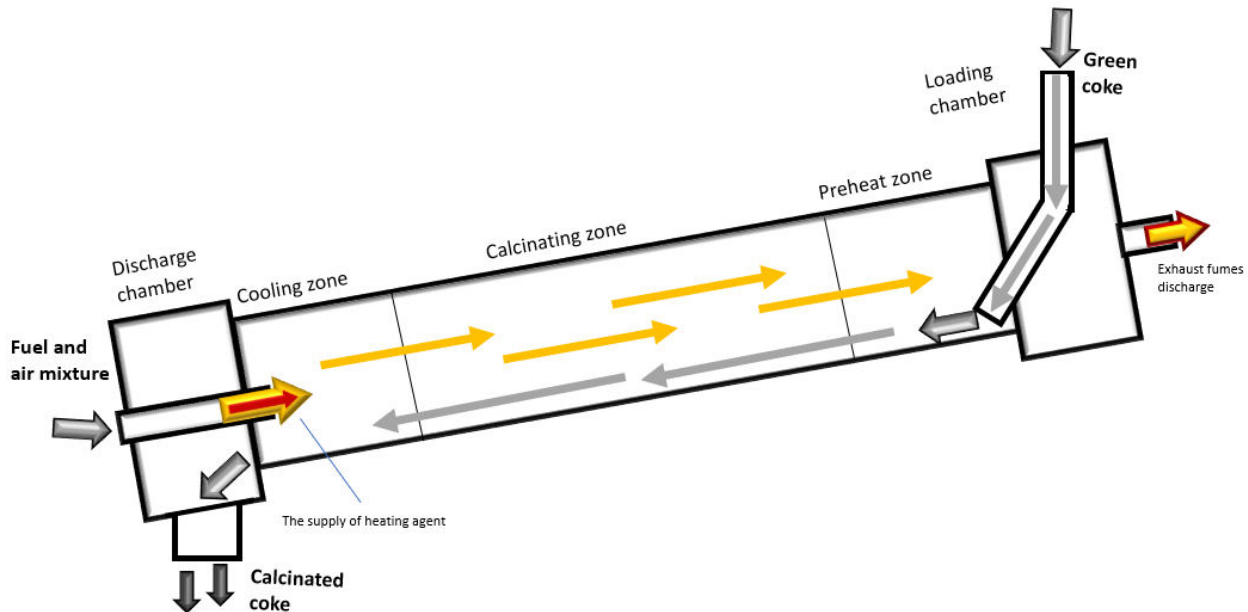


Figure-1. Scheme of coke calcination in a tubular rotary kiln of indirect type.

At present tubular rotary kiln is the best apparatus for obtaining high-quality calcined petcoke at an industrial scale. Green coke is usually calcined in several stages under rather high temperature within the range 1200-1450°C. The ratio of carbon to hydrogen increases to a high extent (from around 20 to 1000 and more) in course of the process. Calcination procedure improves the physical and chemical properties of petcoke which is also purified from residual volatile impurities and some other harmful inclusions (e.g., sulfur compounds).

Calcination procedure in a tubular rotary kiln can be controlled with the automation system. The efficiency factor of petcoke calcination process is usually within 45 to 80 percent depending on the quality of a raw material, furnace parameters and its operating point [10]. One of the key criteria for evaluating the quality of coke calcination is the uniform distribution of the temperature pattern over the entire volume of raw material and inside each individual granule. The temperature pattern here is a set of temperature values at all points of the coke volume under consideration at the moment [9, 11, 12].

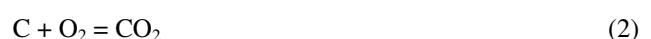
It seems that a universal formalized algorithm for controlling the process of coke calcination has not been found. Depending on the origin of initial oil and the methods of its further processing the patterns for a definite petroleum coke production may differ considerably both for microstructure and properties. For example, with increasing the content of volatile substances in the coke the calcination time should also be increased. If we need to get an isotropic coke the calcination procedure should be performed at higher temperatures than in case of anisotropic coke. The search for a solution to the problem of petroleum coke optimal calcination is one of the areas

of research in oil refinery and metallurgy at the moment [13].

Calcination requires a large consumption of thermal energy as it is an endothermic process. Combustion of volatiles and coke dust at calcination can be considered as an additional source of energy [3, 14, 15].

The proper coke to be used for the production of electrode materials should meet strict requirements. First, it should be rather inert and should not react with any gases and substances. The durability of anode materials will be much higher. The high content of volatile substances, sulfur and metal impurities in initial green coke affects the final calcinated coke porosity. The coefficients of wear resistance and heat resistance are several times lower in porous coke compared to the corresponding coefficients of a proper anodic coke. Metal inclusions in the coke used for anodes production decrease the quality of aluminium being produced.

Reactivity of coke being in direct contact with carbon dioxide and air oxygen is usually evaluated by two laboratory tests. In the first test the reactivity of a coke material is checked directly. The coke samples are placed in a camera with excessive CO₂ content and heated up to the temperature close to 1000°C. The main idea of the second test is the coke reactivity evaluation in contact with air to form carbon monoxide. For this test the anode coke samples are heated to 550°C. Chemical reactions (1) and (2) are performed in course of each test, respectively:





Before testing all samples of coke are weighed, placed in a laboratory furnace that simulates the necessary conditions, and held for some time. After removing the samples from the furnace they are re-weighed. The difference in the samples mass before and after the experiment characterizes the carbon loss. The less mass of a tested sample is lost, the better coke it is considered to be [3, 6, 9, 11].

The quality of a coke depends on the average length of its crystallites L_c . The greater the L_c value is, the higher the quality of a coke should be. The length of crystallites can be determined indirectly through analyzing its function from the heat treatment data. Figure-2 shows generalized function graphs that reflect changes in the length of coke crystallites as a function of the calcination temperature.

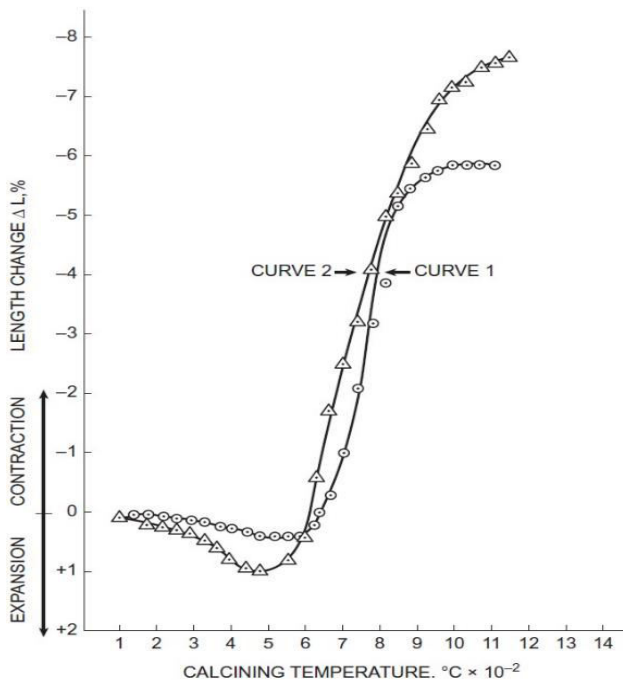


Figure-2. Dimensional changes of anisotropic coke (curve 1); Dimensional changes of isotropic coke (curve 2) [6].

The length of coke grains increases in course of the calcination process according to the formal mechanism of nucleation described by the Avrami equation [6]. Regarding to coke calcination process in a rotary kiln, the equation may be presented in the following form:

$$\Delta H = \frac{H^2 - H^1}{\rho_2 - \rho_1} = \int_{T_1}^{T_2} \frac{d}{dt} (\rho(T)) dT, \quad (3)$$

where H^1 and H^2 are thermal functions of coke and graphite respectively, $T_{1,2}$ – initial and final coke temperature, $\rho_{1,2}$ – density of coke and graphite, $\rho(T)$ – correlation between density and temperature.

There are three crucial parameters that affect the kinetics of coke calcination process: the heating rate and the heating temperature, as well as the holding time (Figure-3). Dependencies of density, porosity and

crystallite size of calcinated coke upon the calcination temperature (each separately) can be described using a third-degree polynomial [6, 16]:

$$Y = a_0 + a_1X + a_2X^2 + a_3X^3. \quad (4)$$

where X is the coke calcination temperature, °C.

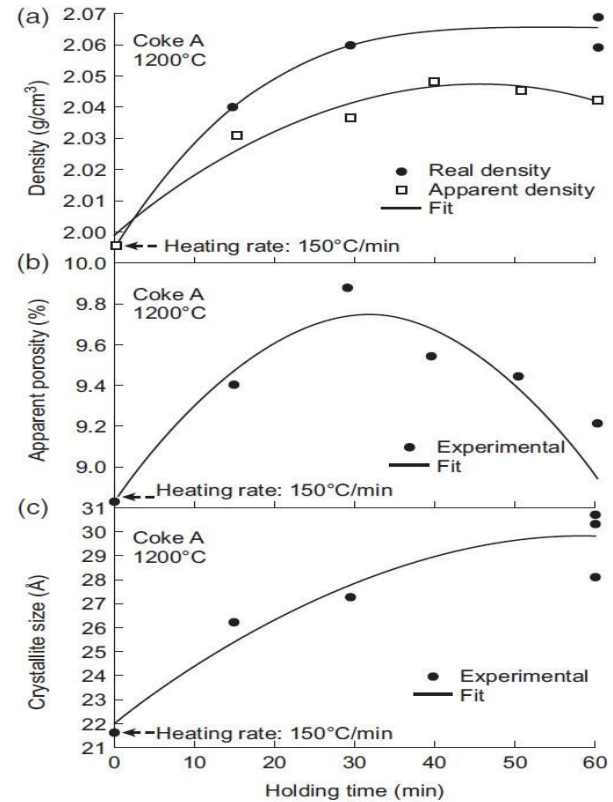


Figure-3. Influence of holding time upon calcined coke properties and quality [6].

During the calcination process, with the growth of maximum calcinating temperature, active growth of coke crystals will occur, porosity will increase and real and apparent densities will also increase respectively. As the heating rate increases, the size of crystals and the actual density decrease, although the apparent density slightly increases. If the optimal heating rate and temperature are determined in the system, the size of crystals and the actual density of a coke will grow with increasing coke exposure time in the furnace. The holding time is one of the most important characteristics of the process, since the change in apparent density and porosity is extreme [6, 9].

RESULTS AND DISCUSSIONS

In order to reliably estimate and predict changes occurring with coke during the calcinating process, it is necessary to refer to the temperature profiles of the furnace used. The temperature profile of a tubular rotary kiln is a graph obtained during the furnace operation that represents the actual temperature changes along the entire



length of the tubular kiln and throughout the entire volume of the processed material [17].

There are no operating furnaces with totally identical input parameters in industry, due to the geometry of a furnace, the quality of green coke and production technology parameters. The efficiency of a coke calcination process is increased with increasing the kiln body length until reaching the maximum value. After that the efficiency is decreased gradually and slightly, hence the production and application of unreasonably long tubular kilns is economically and technologically impractical. Furnaces of different designs are also energy-dependent in various ways; hence they consume different amounts of fuel gas or gasification mixture [18]. Temperature profiles of similar furnaces may differ

slightly from each other. The information contained in the graphs of temperature profiles of different furnaces allows us to determine the optimal length of a tubular rotary kiln. Analysis of these data makes it possible to significantly improve and optimize the process of coke calcination in each individual case [6, 17].

It is possible to evaluate the efficiency of coke purification from some residual impurities using technological graphs that reflect the change in mass concentrations of these compounds in the final product as it moves along the furnace length. Figure 4 presents an approximate graph of the mass fraction transformations for some coke calcination components in the gaseous phase occurring as a result of chemical reactions inside the kiln [19, 20, 21].

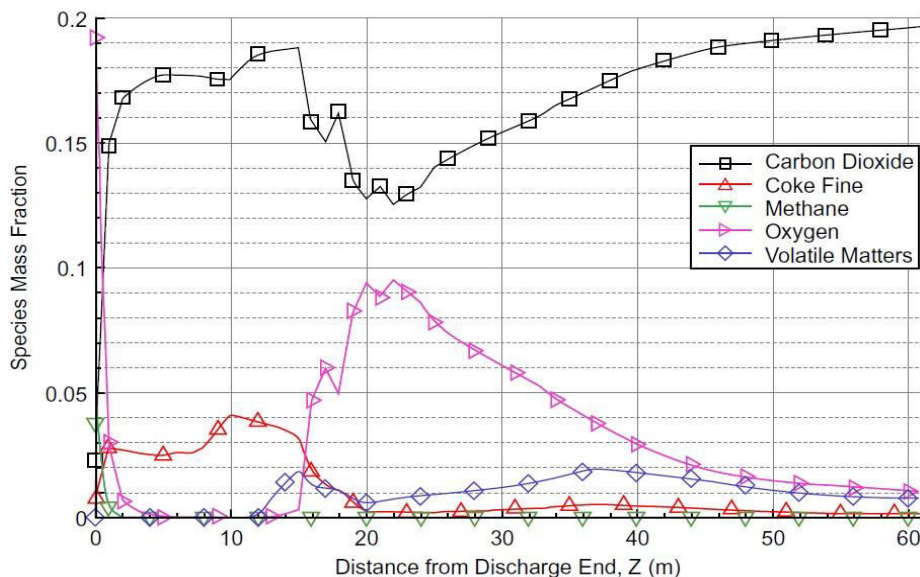


Figure 4. Mass fraction distributions of species in the gaseous phase inside the kiln [21].

Figure-5 shows various temperature profiles along kiln length.

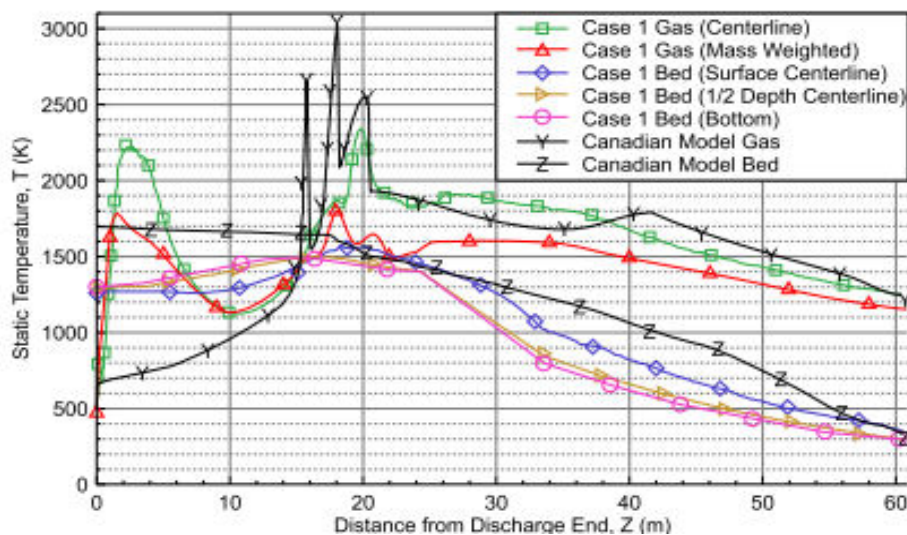


Figure-5. Centerline static temperature profiles for gas and coke beds in the case of tertiary air injection perpendicular to coke bed surface and corresponding mass flow weighted gas temperature.



As coke moves inside the kiln, carbon dioxide is formed and the mass fraction of residual volatile impurities contained in coke gradually decreases. This is the result of burning out the impurities that initially contained in green coke. In order to intensify the main reactions inside the kiln reaction zone it is necessary to provide the sustainable oxygen feed that is being consumed at combustion. In the first temperature zone the coke is warmed up and a slight increase in mass concentrations of volatiles can be observed in the reaction zone. This phenomenon is caused by releasing of these substances from the chemically bound state under the influence of high temperature mainly due to cracking reactions [19, 22]. The release of volatile impurities indicates that the calcination temperature is selected correctly. At the output of the kiln the mass concentrations of oxygen and volatiles should be close to zero. This result would indicate that the active phase of calcination process was performed evenly throughout the entire volume of coke and that the holding time was selected properly [15, 23, 24].

The following technological conditions should be taken into account to ensure satisfactory calcinating results:

- It is necessary to provide an intensive mixing of coke granules with the coolant in the area of volatiles release. Combustion of released volatiles would significantly speed up the heating process of coke being calcinated in the kiln. Changes in the heating rate and an increase in the calcinating temperature both affect the density, porosity and grain size of the calcinated material. It leads to an improvement of the product quality obtained in course of calcination [6, 16, 17].
- Temperature in the reaction zone should reach 1200-1400°C. The design of modern rotary kilns makes it possible to maintain the temperature that ensures the necessary physical and chemical transformations in the coke at half the length of the kiln body. In practice it was proved that the holding time value in such reaction zone is enough for increasing the length of coke grains and providing the optimal grain structure [16, 20, 21].
- Residual volatiles that are contained in a green coke are rich with hydrogen. Water vapor is a noticeable part of the combustion products of the reactions occurring inside the kiln. It can be separated and used in other technological operations. Water vapor is the most common means for quenching and cooling the hot coke. Quenching is necessary to prevent undesirable reactions inside coke volume during the slow cooling process. Water is well known to have an annealing effect on the structure of metal grains at

quenching. There are almost no similar data on the same effect of water upon the coke grains [22, 23, 25].

- Particle size of a green coke to be calcinated should be small enough in order to provide a uniform distribution of concentrations and temperatures inside each coke particle. To consider this phenomenon one should use mathematical modeling of the processes inside a coke particle with taking into account both chemical kinetics and the processes of heat and mass transfer. For a particle of spherical shape this mathematical model is as follows:

$$\frac{\partial T}{\partial \tau} = \frac{\lambda}{dc_p} \left(\frac{\partial^2 T}{\partial r^2} + \frac{2}{r} \frac{\partial T}{\partial r} \right) + \sum_{i=1}^N \frac{w_{ri} \Delta H_i}{dc_p} \quad (1)$$

$$\frac{\partial c_j}{\partial \tau} = D_j \left(\frac{\partial^2 c_j}{\partial r^2} + \frac{2}{r} \frac{\partial c_j}{\partial r} \right) + \sum_{i=1}^N w_{r,j,i} \quad (2)$$

Where:

T	- temperature inside the coke particle, K ;
r	- radius of the coke particle, m ;
λ	- thermal conductivity coefficient, $W \cdot m^{-1} \cdot K^{-1}$;
d	- density, $kg \cdot m^{-3}$;
C_p	- heat capacity, $J \cdot kg^{-1} \cdot K^{-1}$;
$w_{r,j,i}$	- rate of changing an i -th component in the j -th reaction, $kmol \cdot m^{-3} \cdot s^{-1}$;
ΔH_i	- heat effect of the i -th reaction, $kJ \cdot mol^{-1}$;
c_j	- concentration of the j -th component of reaction mixture, $kmol \cdot m^{-3}$;
D_j	- diffusivity, $m^2 \cdot s^{-1}$.
τ	- time, s .

Equations system (4) should be added with the corresponding initial and boundary conditions.

The initial conditions are as follows:

$$c_j(0, r) = \overline{c_j(r)}, j = 1, \dots, 3. \quad T(0, r) = \overline{T(r)}. \quad (6)$$

The boundary conditions are as follows:

$$\begin{aligned} T(\tau, r_0) &= \overline{T(\tau)}; \\ \frac{\partial T(\tau, 0)}{\partial r} &= 0; \\ \frac{\partial c_j(\tau, 0)}{\partial r} &= 0, j = 1, \dots, 3 \\ \frac{\partial T(\tau, r_0)}{\partial r} &= -\alpha(c(\tau, r_0) - C_v); \end{aligned} \quad (7)$$



We used our results of kinetic investigation of a commercial green coke calcination from one of Russian oil refinery plants as kinetic equations for the proposed model. These results can be found elsewhere [26].

To solve the system of equations (5) given above we used the program package *ThermEx* [27, 28]. Temperature profile along the kiln length was used as boundary conditions (see Figure-5).

The main task of modeling was to find the so-called “critical diameter” of a particle that would provide a uniform temperature field inside it. If particles of this size (or smaller) are used, the quality of final coke at the exit from the apparatus will be uniform as well. The modeling results of calcination process in a particle of the critical diameter are presented in Figures 6-8.

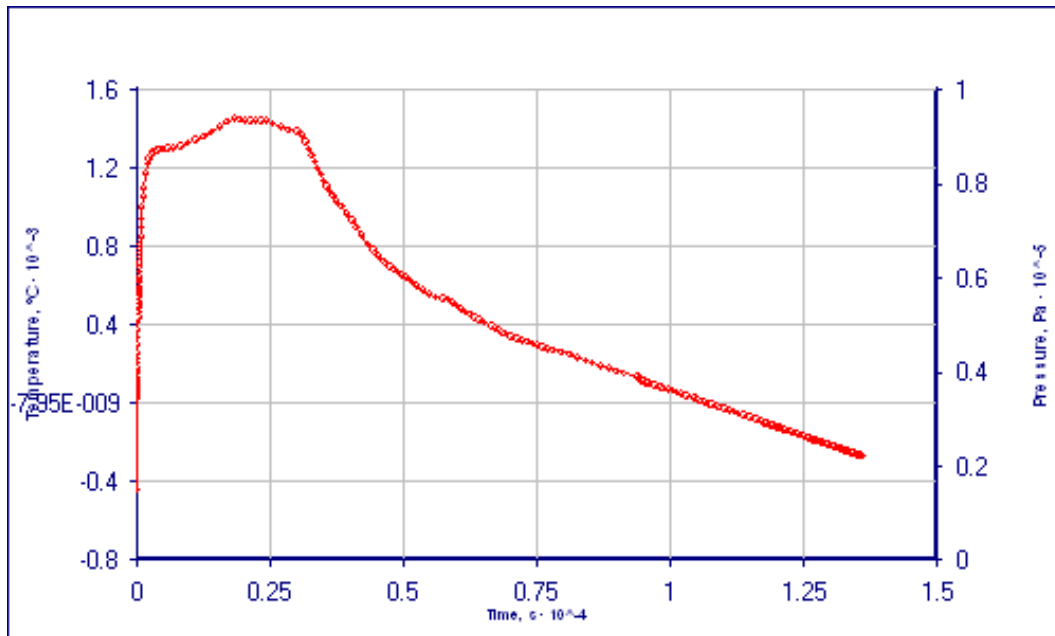


Figure-6. Average temperature of coke particles as a function of residence time in the kiln.

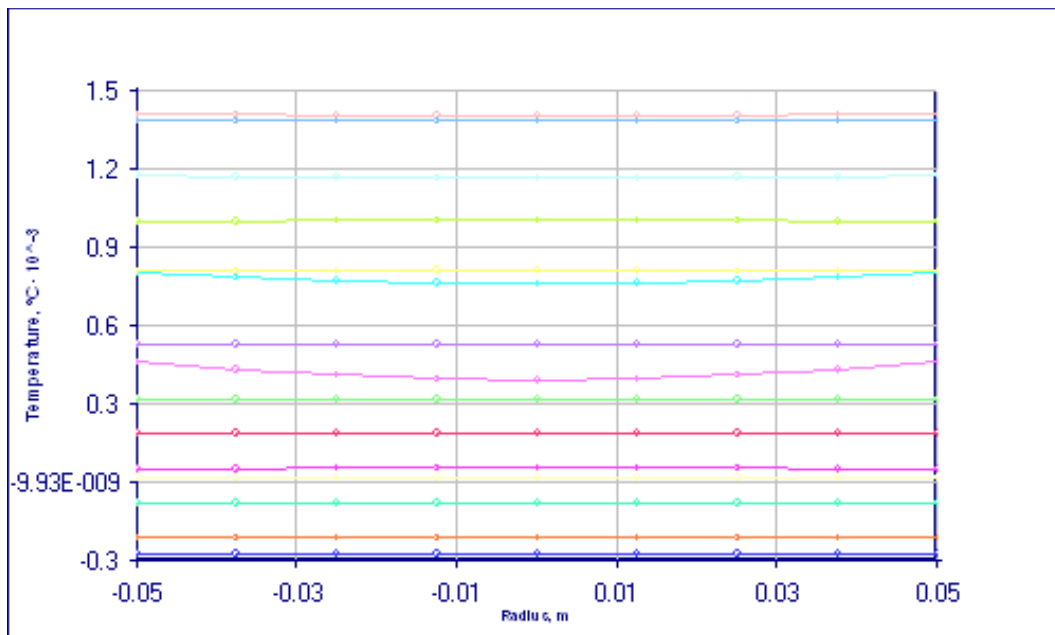


Figure-7. Temperature profile inside a coke particle of 0.05 m radius at calcination.

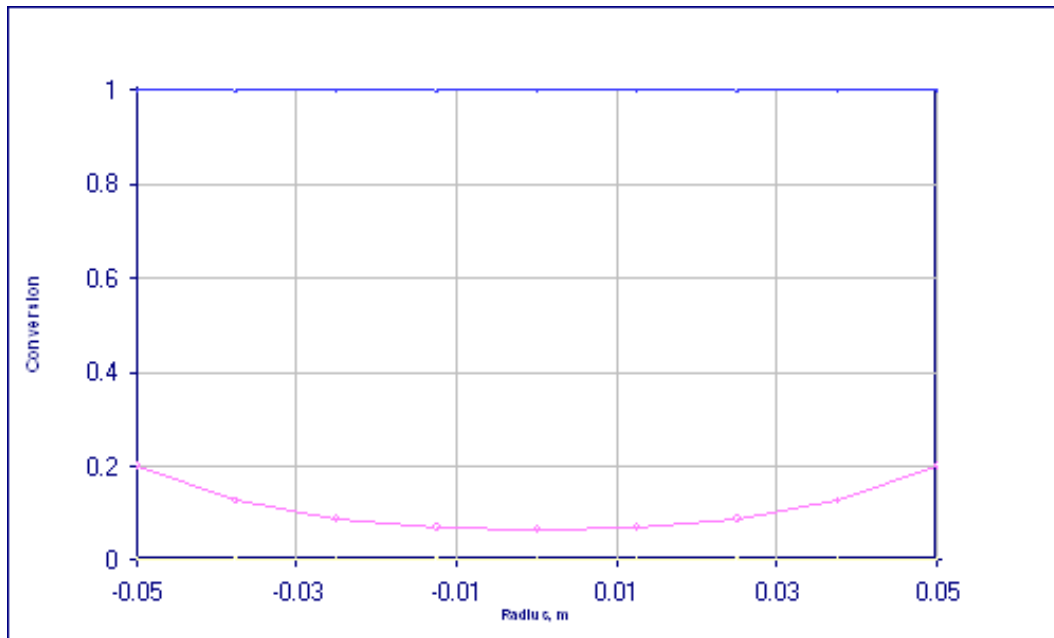


Figure-8. Chemical reactions conversion profile inside a coke particle of 0.05 m radius at calcination.

We claim that it is possible to estimate the maximum allowable coke particle size that should provide a uniform distribution of temperatures and conversions inside the particles of a size or smaller at calcination in the rotary kiln for the given coke batch. It may be done with the proposed mathematical model of the calcination process (see system of equations (5-7)) based on a kinetic model of the definite coke calcination. Under the specified conditions a plug-flow reactor model for the tubular rotary kiln with an ideal mixing approach in every point within each cross-section of coke layer along the kiln length can be applied to describe the coke calcination process. Hence for each coke type it is necessary to carry out a kinetic study using a thermal analyzer (e.g., Labsys evo *SETARAM Instrumentation*, as it was done in [26]), develop a kinetic model of the coke calcination reaction and solve the inverse kinetic task for TG, DTG and DSC experimental responses. After that the critical particle size value for the coke may be calculated with *ThermEx* program package. Next, you can use the process mathematical model to determine the optimal temperature profile and select an appropriate temperature control system in the selected critical points along the tubular rotary kiln length.

Thus, the parameters that affect the coke quality at calcination are: 1) temperature in the hottest zone, 2) reaction zone length and 3) residence time of coke in this zone. The coke quality also depends upon quenching conditions applied to stop the undesirable side reactions. Therefore for developing an optimal control system one should perform a kinetic study and develop a mathematical model of the coke calcination process that also takes into account temperature distribution inside the coke granules at different points along the length of the kiln. This model should be used in the control loop to

generate the necessary control actions that should compensate the incoming disturbances [22].

CONCLUSIONS

- A detailed analysis of the processes occurring at green petroleum coke calcination in a tubular rotary kiln is done. The criterion for producing uniform coke of high quality is formulated.
- The approach to select an optimal technological mode of calcination for a definite green coke in a definite rotary kiln is proposed.
- The main control parameters of petroleum coke calcination process in a tubular rotary kiln are emphasized.
- The requirements for a control system of tubular rotary kiln to run the green coke calcination process under optimal conditions are formulated.

ACKNOWLEDGMENT

Kinetic measurements and modeling were performed in Saint Petersburg Mining University with the equipment and software obtained in course of the Development Program “National Research University” (2011-2013) and the Ministry of Science and Higher Education Program “Support of Fundamental Research Works” (2014-2016, 2017-2019).

The authors are grateful to the Mining University Authorities for creating the conditions, support and attention to the work.



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