THE MAIN NATURAL LOWS OF
HIGH-RATE COAL PYROLYSIS

by

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The importance of coal pyrolysis studies for the development of energy technologies is evident, since pyrolysis is the first stage of any process of coal thermal conversion. In combustion, pyrolysis determines conditions of coal ignition and the rate of char after-burning; in gasification, pyrolysis determines total yield of gasification products. It must be noted that in modern energy technologies pyrolysis occurs at high rate of coal particle heating ($\approx 10^3$ K/s for different fluidized bed, or FB-technologies) or super-high-rate ($\geq 10^5$ K/s for entrained-flow gasification), and in some of them at high pressure.

In CETI during last 12 years the detailed study of pyrolysis in FB laboratory-scale PYROLYSIS-D plant and entrained-flow pilot-scale GSP-01 plant, was carried out.

In this paper main results of mentioned investigations are given. Kinetic constants for bituminous coals and anthracite high heating rates in entrained flow for high temperatures ($\geq 1500\,°C$ and $\geq 1900\,°C$), and in fluidized bed conditions in temperature range 972-1273 K.

In order to describe data obtained in fluidized bed conditions, G-model-based method of calculation of devolatilization dynamics was suited to FB heating conditions.

Calculated and experimental kinetic data are in good agreement. The result proves that at FB-pyrolysis conditions intrinsic mass-transfer limitations are negligible and devolatilization is really kinetic-controlled.

Key words: coal, pyrolysis and gasification kinetic constants, experimetns and modelling

Designers and engineers of energy equipment use to assume that coal particle can be presented as solid porous frame of ash and fixed carbon filled by volatile matter. During rapid pyrolysis, solid frame remains almost intact, but volatiles are released practically instantaneously into intrinsic parietal pores, and then flow out of particle more slowly due to pressure drop [1]. However, these simplified ideas are not incapable to explain a number of experimental facts, and can not be used for development of coal conversion technologies.

On the other hand, coal pyrolysis is investigated in details for the needs of coke chemistry, where processes occur mainly at low heating rate conditions (10^{-2}-10^2 K/s) and at atmospheric pressure. For these conditions a lot of experimental data was obtained (by TGA, DTA, and "heated grid" test methods), allowing to elaborate coal molecular structure and its transformations during pyrolysis [2]. It is established that initial coal macromolecules consist of aromatic fragments binded by heteroatoms (O, N, S) and aliphatic links, and also of light molecular groups weakly connected with aromatic fragments. During heating of coal, thermal destruction takes place, *i. e.* bonds between fragments breakdown and light groups are released in the form of gases. Aromatic fragments-radicals become moveable on this stage, and intermediate metaplastic state of coal takes place. The active bonds of aromatic radical fragments can be stabilised: either by reversible capturing of gaseous molecules, first of all hydrogen or its donors (metastabilisation), or by non-reversible condensation into carbonised coke residue. The smallest (the most moveable) and metastabilised aromatic fragments get out of particles in the form of tars. In the presence of hydrogen or its donors, condensation is inhibited, and this allows to convert the main part of coal matter in liquid state (coal hydropyrolysis and liquefaction). So, neither volatile (gases and tars) nor solid carbon (coke) are present in coal particle initially, but they are formed during pyrolysis, and their yields and properties depend on conditions in which pyrolysis takes place. These ideas are in agreement with the experimentally determined increase of tar yield during rapid heating [3] and increase of total volatile yield during extra-rapid heating [4]. It is also find out that reactivity of coke residue decrease with the increase of when heating rate and final temperature [5].

Several attempts to describe kinetics of pyrolysis are valid only under condition that mass transfer is not the controlling process. Kinetics of decomposition of simple matters can be presented by the expression:

$$\frac{dW_i}{d\tau} = k_i(W_{0i} - W_i)^n \tag{1}$$

where W_{0i} is the final and W_i is the continuous yield of *i*-product, $k_i = k_{0i}\exp(-E_i/RT)$ is rate constant of evolution of *i*-product, E_i and k_{0i} are activation energy and frequency factor for the source of *i*-product (*i*-source), and *n* is reaction order. Expression (1) is often used for processing of experimental data, under assumption that every *i*-product is formed from the single source with activation energy E_i (S-model) and $n = 1$. However, in [2, 6] it is shown that experiments with different heating rates processed by S-model give different values of kinetic constants E_i and k_{0i}. This fact is probably the consequence of the existence of several different bonds surrounding the bond of the *i*-source, that can strengthen or weaken it. The simplest assumption is that every *i*-product is formed by the number of sources having Gaussian distribution of activation energies around E_{0i}-value (G-model) [2]:

$$\frac{dW_{0i}}{dE_i} = \frac{W_{0i}}{\sigma_i\sqrt{2\pi}}\exp\left[-\frac{(E_i - E_{0i})^2}{2\sigma_i^2}\right] \tag{2}$$

In G-model the total yield of the *i*-product (per dry ash free – daf coal matter) is assumed to be constant for given coal and is defined theoretically as integral of expression (2). Experimentally the total yield W_{0i}, of the *i*-product, G-model kinetic constants E_i, k_{0i}, and σ_i can be determined by double differentiation of the evolution curves obtained for at least 4 different heating rates 6 . In 2 the generalised values of W_{0i}, E_i, k_{0i}, and σ_i are determined for gaseous and liquid products for different coals at constant heating rates, which are in good agreement with the experiments.

However, for high and super-high heating rates and at high pressure all above-mentioned ideas have not yet been approved due to the lack of experimental data. Moreover, for FB-pyrolysis up to the moment it was considered that the errors connected with gas transport and mixing did not allow to carry out kinetic measuring at all. It is believed that non-linear particle temperature change impede application of G-model--based calculations, and that all kinetic-based calculations can not be correct if the in-pore mass transfer is not taken into account. The evident Quantitative dependence of the char reactivity on temperature during FB-pyroly- sis and or fixed carbon conversion at extra--rapid heating in entrained-flow gasification are not yet obtained, also.

In the Coal Energy Technology Insti- tute (CETI) during last 12 years the detailed study of pyrolysis in FB (laboratory-scale PY- ROLYSIS-D plant at 0.1-2.5 MPa pressure and pilot-scale UTT-2-2.76 plant[1] with coal feed rate 100-500 kg/h) and entrained-flow gasification technologies (pilot-scale GSP-0.1 plant[2] with coal feed rate 50-150 kg/h), was car- ried out. The main results obtained are the fol- lowing.

1. In entrained-flow gasification with ther- mal shock (heating rate $\geq 10^6$ K/s), and final temperature ≥ 1500 °C for bitumi- nous coals and ≥ 1900 °C for anthracite the degree of carbon conversion X_c dur- ing pyrolysis (0.1-0.2 s) reaches 90-95% 7 (fig. 1). In open literature there is no results obtained under the mentioned conditions. Results are in good agree- ment with above-mentioned idea pro- posed for less rigid conditions that solid carbon yield increase with the increase
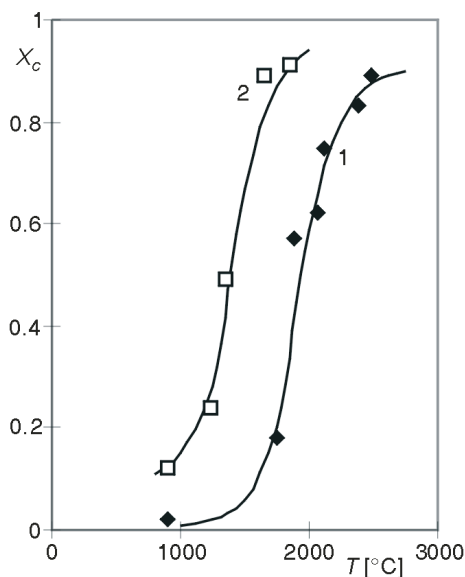

Figure 1. X_c dependence on T when entrained flow gasification $(\tau \leq 0.1$-0.2 s, $P = 0.1$ MPa) of dry pulverized Donetsk coals
1 – anthracite ($A_d = 31\%$, $V_{daf} = 6\%$),
2 – bituminous ($A_d = 20$-31%, $V_{daf} = 44\%$)

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[1]   The test plant was constructed and some test were carried out in co-operation with *Pivdentechenergo* (Lviv, Ukraine), namely with B. Sinyakevich and Z. Geletiy

[2]   The test plant was constructed and some tests were carried out in co-operation with *ITEN Energoinvest* (former Yugoslavia) and IVTAN (Russia), namely with M. Sijerčić, S. Neuman, A. Pyatenko *etc*.

of heating rate and final temperature increase. It must be noted that at high temperature and in presence of CO_2 and H_2O all tars convert homogeneously to CO and H_2. This allows to develop 2-stage entrained-flow gasification technologies with reduced coal particles residence time [8].

2. For anthracite, the decrease of the coke residue reactivity in FB-pyrolysis with the increase of temperature T_b from 973 up to 1273 K established in [9] can be described by expression:

$$k_0(T) = k_{0(max)} \left[1 - 1.01 \cdot 10^7 \exp \frac{14472}{T_b} \right] \tag{3}$$

where k_0 is frequency factor in Arrhenius expression for coke residue – air oxygen reaction rate constant $k = k_0 \exp(-E_a/RT)$ [10]. It seems to be the first reliable quantitative presentation of ideas about increase of coke carbonisation degree with the increase of heating rate and final temperature [2, 5]. For bituminous coals this factor is negligible comparing to external diffusion combustion rate limitation, but in FB-pyrolysis the effect particles agglomeration (mainly for the particles with ≤ 0.25 mm in size) takes place (fig. 2). It is believed that agglomeration is a consequence of metaplastic properties of coal matter during pyrolysis.
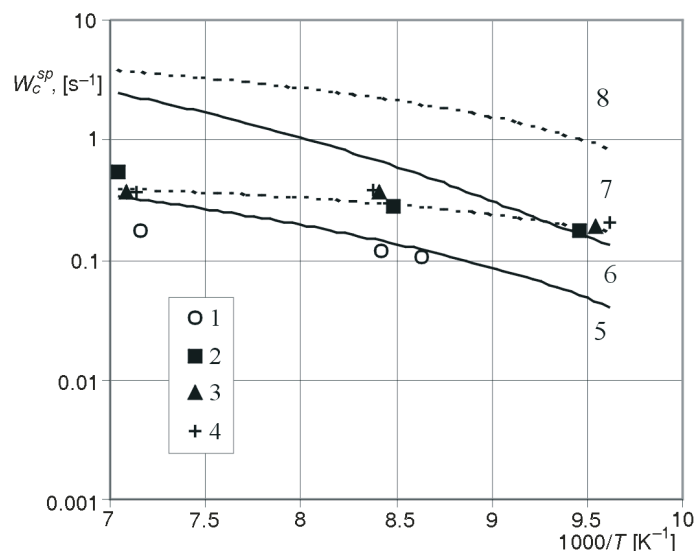

Figure 2. Specific combustion rate W_c^{sp} dependence on T_b when bituminous coal ($A_d = 8\%$, $V_{daf} = 44\%$) pyrolysis/combustion in FB without intermediate interruption. Experimental data (initial particle size 0.06-0.1 mm)
1 – 0.1 MPa, 2 – 0.8 MPa, 3 – 1.0 MPa, 4 – 1.2 MPa; calculated dependences: 5, 6 – 0.1 MPa, 7, 8 – 0.8 to 1.2 MPa; 6, 8 – medium size 0.08 mm, 5, 7 – medium size 0.26 mm

3. In FB at T_b = 750-1000 °C, P = 0.1-2.5 MPa the coal particles with ≤2.5 mm in size, are heated up to bed temperature within 0.2-4.0 s, so the main part of devolatilization occurs at isothermal conditions at bed temperature. Gaseous volatiles evolution depends on the conditions of their evacuation from the particle. If the fluidization velocity is low and the concentration of products around the particles is noticeable, the rate of gaseous volatile evolution is limited, most probably the reverse reactions of gaseous products with metaplastic coal matter takes place (the reactions of metastabilization of the radical fragments of coal macromolecules). This results in limitation of H_2, CH_4, and CO evolution rates by some top (equilibrium) partial pressures P_{0i} of gas products in the form of:

$$\frac{dW_i}{d\tau} = k_{1i}(W_{0i} - W_i) - k_{2i}P_iW_{0i} \qquad (4)$$

where P_i is partial pressure of the current i-product, or, accounting $P_{0i} = k_{1i}/k_{2i}$ ratio between the rate constants of direct k_{1i} and reverse k_{2i} reactions of pyrolysis gas evolution, in the form of:

$$\frac{dW_i}{d\tau} = k_{1i}\left(1 - \frac{k_{1i}W_{0i}P}{G_{gc}P_{0i}}\right)^1(W_{0i} - W_i) \qquad (5)$$

at high pressure P in reaction zone ($P \gg P_{0i}$) [9, 11]. Here G_{gc} is the flow of carrier gas which was inert or CO_2 in tests. The detected top partial pressures P_{0i} of gas products increase (Arrhenius-like) with temperature (fig. 3), very slightly depend on coal type and do not depend on total pressure (when $P > P_{0i}$) and on particle size. Therefore, it would be impossible to obtain medium-calorific (non-diluted) pyrolysis gas at $P \geq 10$ MPa in FB. If preliminary pyrolysis is organized by using some
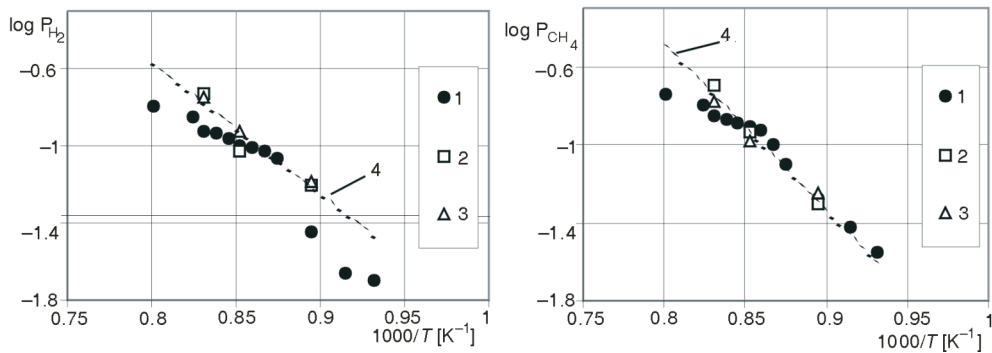
Figure 3. Top partial pressure P_{0i} dependence of T_b for H_2 and CH_4 when FB-pyrolysis of Donetsk (A_d = 45%, V_{daf} = 42%) and British (A_d = 5%, V_{daf} = 39%) bituminous coals of size from 04.-0.6 mm to 1.6-2.5 mm (P = 1.0-2.5 MPa)
1 – *direct experimental data obtained at low G_{gc} and big coal samples, 2, 3 – values calculated from experimental data at medium G_{gc} and different coal samples (accordingly by k_i decrease and by maximum P_i values), 4 – Arrhenius generalization of calculated values*

solid heat-carrier (for instance, in two-stage pressurized circulating fluidized bed (PCFB) technologies, some carrier gas flow through pyrolyzer is necessary 12 , and the rate of devolatilization would be proportional to G_{gc} and to P_{0i}.

4. In FB in the same ranges of temperature, pressure and particle size, but at high fluidization velocity and low concentration of products around the particle, the rate of gas evolution from coal increases during the period of particle heating up to bed temperature and then decreases in the form of expression (1) ($n = 1$). As yields and rate constants of gaseous products evolution determined experimentally increase (the last – Arrhenius-like) with bed temperature (tab. 1) and do not depend on pressure, fluidization velocity and particle size 9, 11, 13 , process is kinetically controlled. It was possible to obtain this result only by using method of data processing which takes into account gas transport and gas mixing in FB-reactor and to elucidate only influence of chemical kinetics.

Unlike results presented at the items 1, and 3 which are confirmed on pilot-scale plants, and results presented at the item 2 which have merely chemical ground, results obtained in FB-experiments (item 4) have to be checked by comparison with generalized kinetic data based on G-model 2 (accounting that all another known kinetic data obtained in just another conditions were processed by S-model and therefore would not be used).

In particular, the question is what is the contribution of intrinsic mass transfer and why in FB-experiments gas-products yields increase with the increase of bed temperature in spite of G-model's assumption about the constant products' yields. It must be taken into account that FB-experiments really cannot be processed by G-model, because it is not possible to change heating rate and the fact that change of particle temperature is not linear. That's why the only way to check FB data is to suit the G-model-based method of calculation of devolatilization dynamics to FB heating conditions using generalized kinetic data of 2 and to compare the experimental and calculated data, at least for H_2 as for one of the major products.

The algorithm of such method was developed in CETI and is presented at fig. 4. It includes calculation of combined convection-conduction FB heat-exchange coefficient

Table 1. The yields and rate constants of the main gaseous products of Donetsk bituminous coal pyrolysis in FB ($P = 0,1$-$2,5$ MPa, particle size $0,4$-$0,6…1,6$-$2,5$ mm)

T_b, [K]	Rate constants k_{1i}, [s^{-1}]				Product yields, [10^{-3} nm^3 kg^{-1}]$_{daf}$				Element yields, [% mas.]$_{daf}$		
	H_2	CH_4	CO	CO_2	H_2	CH_4	CO	CO_2	C	H	O
1023	0.30±0.05	0.8±0.1	0.5±0.2	0.9±0.1	60±15	45±12	60±15	15±5	6.0	1.2	6.0
1073	0.37±0.06	0.9±0.1	0.7±0.2	1.0±0.1	80±25	50±16	70±25	20±8	7.0	1.5	7.4
1113	0.43±0.08	1.0±0.2	0.7±0.3	1.3±0.3	115±26	58±18	80±22	28±10	8.3	2.0	9.0
1153	0.55±0.05	1.4±0.2	1.0±0.2	1.7±0.3	150±25	66±16	90±20	30±12	9.3	2.3	10.0
1193	0.63±0.06	1.6±0.3	1.4±0.4	1.9±0.5	180±30	75±20	95±22	30±14	10.0	2.7	10.4
1233	0.95±0.06	1.9±0.4	1.7±0.5	–	210±25	75±18	95±20	30±12	10.0	3.0	10.4
1253	1.10±0.08	–	–	–	225±30	70±22	95±25	30±16	9.7	3.1	10.4
Initial volatile elementary composition									18.3	5.6	11.8

Initial data

W_0, K_0, E_0, σ by [2], ΔE set

Ar, Pr, λ_g, ρ_p, c_p by literature, T_b, d_p, d_b, Δ_t, set

Calculation of the i-source gas evolution

Calculation of $\left.\dfrac{dW_0}{dE}\right|_{E_i}$ by expression:

$$\frac{dW_0}{dE} = \frac{W_0}{\sigma\sqrt{2\pi}}\exp\left(-\frac{(E_i - E_0)^2}{2\sigma^2}\right),$$

$$W_{0i} = \left.\frac{dW_0}{dE}\right|_{E_i}\Delta E$$

Calculation of α in FB by expressions:

$$\alpha = \frac{\mathrm{Nu}\ \lambda_g}{d_b}$$

$$\mathrm{Nu} = \mathrm{Nu}_{max} - (\mathrm{Nu}_1 - \mathrm{Nu}_{max})\exp\left(-\frac{d_p}{4d_b}\right)$$

$$\mathrm{Nu}_1 = 10 + 0.23(\mathrm{Ar}\cdot\mathrm{Pr})^{1/3}$$

$$\mathrm{Nu}_{max} = 0.85\mathrm{Ar}^{0.19} + 0.006\mathrm{Ar}^{0.5}\mathrm{Pr}^{0.33}$$

Calculation of $W_i(\tau)$ by expression:

$$W_i = W_{0i}\left[1 - \exp\left(-k_0\int_0^{\tau}\exp\left(-\frac{E_i}{RT_p(\tau)}\right)d\tau\right)\right]$$

Calculation of $\dfrac{dW_i}{dt}(\tau)$ by $W_i(\tau)$ differentiation

Calculation of $T_p(\tau)$ by expression:

$$T_p = T_b - (T_b - 293)\exp\left(-\frac{\alpha F_p}{m_p c_p \left(1 + \frac{\alpha d_b}{10\lambda_p}\right)}\tau\right)$$

Calculation of $W(\tau) = \sum_i W_i(\tau)$

Calculation of $dW/d\tau(\tau)$ by differentiation

Calculation of $W_i(\tau)$ for every i source

Calculation of $\dfrac{dW}{d\tau}(W)$, defining the resulting rate constant $\bar{k}$ as tangent of linear section inclination angle
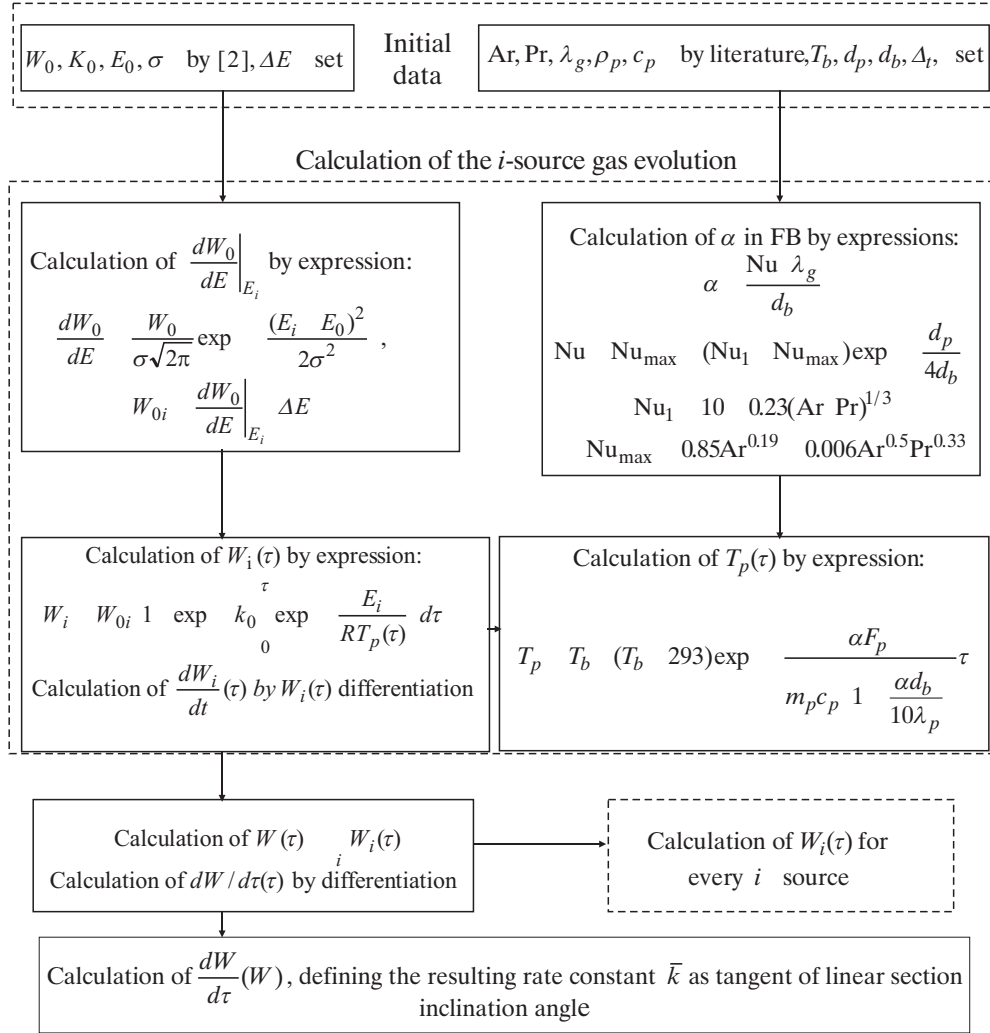
Figure 4. Algorithm of G-model-based method of calculation of devolatilization dynamics at FB heating conditions

depending on gas type, temperature, pressure, size of coal d_p and bed particles d_b, and calculation of coal particle temperature change $T_p(\tau)$. In the first approach endothermic thermodestruction specific heat (400-600 kJ/kg when $T_p = 700$-900 K) can be accounted by 30-50% decreasing of α value. The next step is presentation of Gauss distribution (2) as histogram using for H_2 $k_0 = 1\cdot10^{14}$ s^{-1}, $E_a = 336{,}7$ kJ/mol, $\sigma = 49{,}9$ kJ/mol [2], $W_0 = 225\cdot10^{-3}$ nm^3/kg daf [11], and histogram step $\Delta E < 0{,}1$. For every i-element of histogram (i-source with $E = E_i$) function $W_i(\tau)$ is calculated numerically (time step $\Delta\tau \le 0{,}2$ s) using the expression:

$$W_i = W_{0i} \left[1 - \exp \left(-k_0 \int_0^\tau \exp \left(-\frac{E_i}{RT_p(\tau)} \right) d\tau \right) \right] \qquad (6)$$

which is the solution of expression (1) (n = 1) accounting temperature change $T_p(\tau)$. Numerical differentiation of $W_i(\tau)$ gives $dW_i/d\tau(\tau)$, and summing up $W_i(\tau)$ and $dW_i/d\tau(\tau)$ of all i-sources – gives the final result.

Figure 5 presents calculated $T_p(\tau)$, $W(\tau)$, and $dW/d\tau(\tau)$ in comparison with experimental data obtained for Donetsk bituminous coal at $P = 0,1$ MPa, $T_b = 1200$ K. For different T_b calculated $dW/d\tau(W)$ is presented on fig. 6. It can be mentioned that:

– calculated and experimental $dW/d\tau$ (τ) are in rather good agreement,
– decreasing parts of calculated $dW/d\tau(W)$ curves have long linear sections what allow to define for them rate constant values by processing in the form of expression (1), the same is valid for experimental data,
– calculated values of the rate constant k and of W_0 (the last can be defined as W value at the point where $dW/d\tau$ decreases down to 10^{-6} nm^3/s, or to gas analyzer sensibility limit) increase with bed temperature as well as experimental ones.
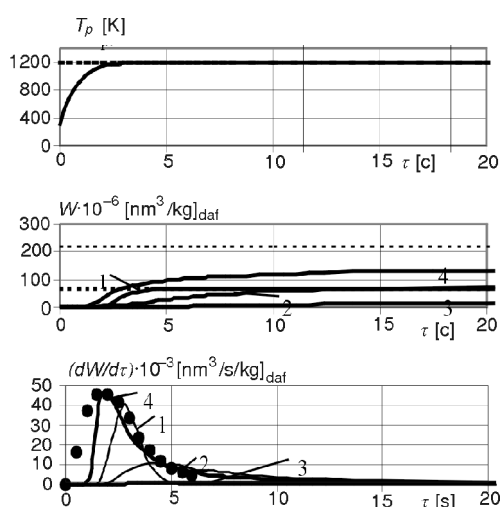

Figure 5. Time dependences, calculated by G-model based method of T_p, W, and $dW/d\tau$ for H$_2$ for FB-pyrolysis of Donetsk bituminous coal; $P = 0.1$ MPa, $T_b = 1200$ K, particle size 1.0-1.6 mm, $W_0 = 225 \cdot 10^{-3}$ nm^3/kg $_{daf}$ (exp); $E_0 = 336.7$ kJ/mol, $\sigma = 49.9$ kJ/mol; $k_0 = 1 \cdot 10^{14}$ s^{-1} [2]
1, 2, 3 – $\Delta E = 4$ kJ/mol, 1 – $E_i = E_0 - 0.5 \sigma$, 2 – $E_i = E_0$, 3 – $E_i = E_0 + 0.5 s$, 4 – experimental data, averaged from different sources
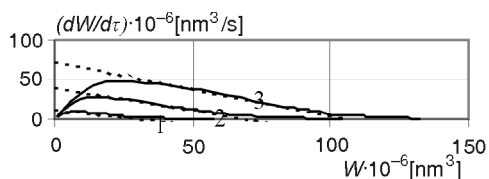

Figure 6. $DW/d\tau$ dependence on W calculated by G-model-based method for H$_2$ for FB-pyroly- sis of Donetsk bituminous coal at different T_b
1 – 1023 K (k = 0.36 s^{-1}), 2 – 1123 K (k = 0.56 s^{-1}), 3 – 1200 K (k = 0.67 s^{-1})
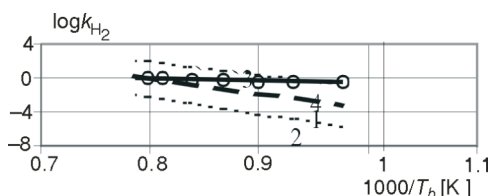

Figure 7. Comparison of calculated and experimental Arrhenius dependences for bituminous coal
1, 2, 3 – direct S-model ap- proximation using G-model data from [2];
1 – $E_i = E_0$, 2 – $E_i = E_0 - \sigma$, 3 – $E_i = E_0 + \sigma$,
4 – approximation of G-model-based method results for FB-pyrolysis; points – experimental data of FB-pyrolysis

On fig. 7 calculated and experimental $k(T_b)$ are compared showing perfect coincidence. It is important to note that the result of calculation is obtained using G-model kinetic only, with no accounting of mass-transfer limitations. This proves that at FB-pyrolysis conditions intrinsic mass-transfer limitations are negligible and devolatilization is really kinetic-controlled. The applied importance of this result is that in practical applications tar and another products evolution which are difficult to detect directly in FB can be calculated in the same way, *i. e.* by using the G-model-based method proposed above.

The main conclusion on CETI pyrolysis studies is that for development of the modern coal conversion technologies simplified "mechanical" presentation of pyrolysis process can not be used but modern "macromolecular" due to much better capability of the last to explain experimental facts. All above-mentioned results seem to be important for development of the new efficient coal conversion technologies based on different kinds of FB and entrained flow regimes.

Acknowledgments

Nomenclature

A – ash content, [%]
Ar   – Archimedes number, [–]
c_p – specific heat of coal, [J/kgK]
d_b – bed particle diameter, [m]
d_p – coal particle diameter, [m]
E – activation energy of pyrolysis gas evolution, [kJ/mol]
E_0 – most probable activation energy, [kJ/mol]
F_p – coal particle surface, [m^2]
G_{gc} – flow of carrier gas, [kg/s]
k – rate constant of pyrolysis gas evolution, [s^{-1}]
k_0 – frequency factor, [s^{-1}]
k_1 – direct rate constant of pyrolysis gas evolution, [s^{-1}]
k_2 – reverse rate constant of pyrolysis gas evolution, [s^{-1}]
m_p – mass of coal particle, [kg]
Nu   – Nusselt number, [–]
n – reaction order
P – pressure, [MPa]
P_i – ratio of direct and reverse rate constants of gas evolution ($=k_{1i}/k_{2i}$)
P_0 – partial pressure of gas products, [MPa]
Pr   – Prandtl number, [–]

R – universal gas constant, [J/mol·K]
T – temperature, [K]
T_b – fluidized bed temperature, [K]
T_p – coal particle temperature, [K]
V – volatile content, [%]
W – yield of pyrolysis gas evolution for time τ, [nm^3/kg]
W_0 – final yield of pyrolysis gas evoluation, [nm^3/kg]
W_c^{sp} – specific combustion rate of coal, [s^{-1}]
X_C – degree of carbon conversion, [–]

Greek letters

α – heat transfer coefficient, [W/m^{2}K]
ΔE – difference of activation energies, [kJ/mol]
λ_g – gas conductivity, [J/mK]
λ_p – conductivity of coal, [J/mK]
ρ_p – coal particle density, [kg/m^3]
σ – standard deviation of activation energies, [kJ/mol]
τ – time, [s]

Subscripts

d – dry
daf   – dry ach free
i – related to the i-product of pyrolysis

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