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Mechanically assisted low-temperature pyrolysis of hydrocarbons

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We report experimental setups and conditions leading to pyrolysis (cracking) of such gaseous hydrocarbons as methane, mixed propane and butane, at the temperatures of the heater below 200°C. The process was mechanically assisted by putting the substances being decomposed into a dynamic interaction with the tin and bismuth alloy. The alloy had periodically changing phase state thus creating fractal interfaces between its surface and the gases. Interaction of the gases with mechanically produced fractal surfaces of the alloy made possible gas decomposition even at lower temperatures of the heater (150°C). At this temperature the heater couldn’t melt the alloy in the heated volume with the gas.

**I. INTRODUCTION**

Technologically innovative processing of fuel types such as black oil and gaseous hydrocarbons is of valuable interest to the petrochemical industry with respect to a better energy efficiency. Also refining and pyrolysis of liquid and gaseous hydrocarbons belong to the line

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of socially important industries with respect to the value of their final fuel products in the forms of light hydrocarbons, hydrogen and carbon.

One may note that to obtain light hydrocarbons in fluidized bed reactors, the raw biomass requires an extensive mechanical processing to maximize the reactive surface of its particles and to minimize the temperature of their chemical processing [1]. In addition to this, the addition of catalytic particles to such reactors contributes to increase of their energy efficiency by minimization of heat losses. These are unavoidable in high temperature applications, compared to when using catalysts.

Additions of catalysts to reactors may take place in the form of dopants of non-reactive porous particles. In such a case an additional enhancement of the chemical reactivity takes place because of the increased possibilities for reactants to reach the catalyst on the large area of the fractal surfaces in the pores. An example of an analysis of special reactive diffusion regimes on fractal catalysts is presented in [3]. Highly developed fractal geometries of internal surfaces of the pores in the particles with catalysts is beneficial for a chemical productivity and an energy efficiency of such fluidized bed reactors, as was reported in [2].

In opposite to the fractal based enhancement of the catalytic chemical mechanisms working in fluidized bed reactors, one may consider purely physical mechanisms working in reactors [6, 7]. Accordingly to our understanding of internal mechanisms working in these reactors (where melts of non-reacting alloys of tin and bismuth are applied), it is possible to increase the rate of pyrolysis of the hydrocarbons using dynamic interactions between the alloys and hydrocarbons on their fractal interfaces. These interfaces dynamically appear, change and disappear during the motion of the hydrocarbons via the alloys. One may also create them mechanically at temperatures below the solidification temperature of the alloys by using fracturing [5] methods. In the first case, the alloys are under-cooled by a convective heat transfer as is described in paper [8]. Such under-cooling creates disordered dendritic fractal-like structures between different phases in the alloys. Also, a dynamic solidification of these structures creates fractal interfaces between them and the cooling gas. The solidification temperature of the eutectic Sn:Bi alloy is $138^\circ C$. The cooling gas has to have even lower temperatures. Because it appeared possible to experimentally decompose this gas during convective heat transfer from the alloy, the authors of [6] initiated a public discussion at an Internet forum (which nowadays is inaccessible) about their experimental results in 2005. The experiments showed a possibility for methane, which was chosen as a cooling gas, to be
decomposed at temperatures below 200°C. Accordingly to the thermodynamic calculations in [4], the minimal temperature for the start of a methane thermal decomposition is 327°C.

Extensive public discussions about the experiments with a substantially lower temperature of a methane decomposition did not result in a physical explanation of these experimental results. There were no mentioning about fractal interfaces between the alloy and hydrocarbons during the discussions. However, the presence and possible role of these interfaces was noted recently due to the analysis of the papers [8–13]. Also, the researchers were inspired by the successful experiments aiming reproducing the results of the authors [6, 7]. The current paper focuses on the physical parameters and mechanisms of pyrolysis of gaseous hydrocarbons, which takes place due to their motion along the interface of the alloys.

II. EXPERIMENTAL

As one may assume from the introduction, the initial working hypothesis on the physical mechanisms of pyrolysis of gaseous hydrocarbons at extremely low temperatures has its basis in the presence of fractal interfaces between a gas and an alloy. Thus, the experiments were conducted with this idea in mind. To explore the effects of the dynamic creation of fractal interfaces between the alloy and the gas due to convective cooling of the alloy by the moving gas, the following three experimental setups were made.

II.1. Under-cooling of the alloy using setup in FIG.1 takes places during the motion of gas bubbles through the top part of the molten alloy in the curved glass tube. The gas inside the bubbles is initially at room temperature. Its passage via the alloy rises its temperature up to a value in the range of 95 − 100°C. Such temperatures were measured using a thermocouple on the output part of a test tube with an accuracy of +/− 1°C. The time for a bubble to pass via the alloy is lower than one second. This time is enough for part of the gas in a bubble to be decomposed and to create carbon particles. Some of these particles fall and float on the surface of the alloy and form a black deposition moving with the alloy during bubbling. A photo of this deposition taken after solidification of the alloy are presented in the Results and Discussion section. Some carbon particles get trapped inside the alloy during a passage of the bubble. These particles form porous structures inside the alloy, which presumably include hydrogen as secondary product of hydrocarbons’
decomposition upon solidification of the whole alloy.

**FIG. 1:** The first experimental setup: 1 - volume containing propane/butane mixture; 2 - valve to regulate flow of gas; 3,4,5 - thermocouples; 6 - heating wire; 7 - U-shape test glass with Sn:Bi alloy; 8 - volume with water; 9 - output tube

**II.2.** The experimental setup in FIG.2 partially reproduces the setup, which was discussed publicly in Internet by the authors of [6]. It differs by the addition of a circular loop for the gas, which after an initial filling of the tubes and test bulb is pumped by a membrane pump in a loop. A magnet (8) was used to mix the gas and alloy to obtain a better under-cooling of the alloy to produce as much carbon particles and hydrogen as possible. Because of the use of a cooler, which was made of approximately a 3 m copper tube submerged in a cold water, the alloy was under-cooled too efficiently. It was only 15 g of the alloy in the bulb. Due to this, the rotation of the magnet was stopped and the alloy was periodically melted and solidified due to a periodic switching of the pump in an off and on mode. The measured temperature of the gas in the glass bubble was 90°C, while the alloy was in a molten stage. The pump was switched off when the temperature of the gas was decreased to a temperature of 85°C and the alloy became solid. It is necessary to note, that the end of the thermocouple was not submerged in the alloy. Instead the temperature was measured at some distance from its surface. Because of trapping of carbon particles to the alloy being solidified, its melting took place slower than it could have done without the particles. Due to the strong pulsating blowing of the gas being pumped by the membrane pump, the boundary part of the alloy often became separated from its main part up to the end of blowing cycle. The maximum temperature of the heater was 250°C. Also the experiments included measurements of the gas composition with an Agilent Technologies 490 Micro GC.
FIG. 2: The second experimental setup: 1 - Sn:Bi alloy; 2 - glass bulb; 3 - cooler; 4 - filter; 5 - intermediate flexible volume; 6 - membrane pump; 7 - thermometer with K-type thermocouple attached; 8 - magnet in a plastic cover; 9 - thermo-stabilized heater with magnetic mixer

An important note was made during observation of the dynamics in the bulb before a melting of the alloy took place. This note is described in more detail below. It yielded a modification of the experimental setup accordingly FIG.3.

The alloy was prepared in the form of chopped sticks of a wire with a diameter 2.5 mm. The sticks were chopped in such a way, that their ends were stretched and broken during the process. In such a case, the surface of a broken alloy acquires a fractal-like geometry [5].

The sticks were mixed by a magnet during the initial step-by-step heating of a bulb. The gas was also pumped during this heating. It was noted during observations of this process that tiny black bars appeared among the alloy sticks. The length of the bars was comparable with the diameter of the chopped wire sticks of the alloy. These tiny black bars disappeared when the motion of a magnet and pumping of the gas was switched off and a melting of the alloy was achieved. However, their appearance was noted and hypothetically linked to the existence of fractal surfaces on the ends of the chopped alloy sticks.

II.3. To test the hypothesis about decomposition of butane/propane mixture due to its motion along the fractal surfaces on the ends of the sticks, the experimental setup in
FIG. 3 was made. The mixing of the sticks by a magnet and their melting were not assumed during the experiments using this setup. These experiments were conducted at a 150°C temperature of the heater and a maximum temperature of the gas in a glass bulb of 70°C. Some of the alloy sticks were constantly moving and colliding under the pulsating flow of the gas, coming from the membrane pump.

![Diagram](image.png)

FIG. 3: The third experimental setup: 1 - Sn:Bi alloy sticks; 2 - glass bulb; 3 - cooler; 4 - filter; 5 - intermediate flexible volume; 6 - membrane pump; 7 - thermometer with K-type thermocouple attached; 8 - thermo-stabilized heater

III. RESULTS AND DISCUSSION

A. Results of the experiments using the first experimental setup

The experiments using setup in FIG.1 were conducted using two different alloys. The first one has a 57% Sn and 43% Bi composition with an eutectic temperature of 138°C. The second alloy consisted of 54% Pb, 11% Sn and 35% Bi. It has a 140°C melting temperature. Accordingly to [14], inclusion of lead to the alloy gives much more complicated structures of different phases in different conditions in comparison to an eutectic Sn:Bi alloy.

The alloy with lead showed a lower efficiency in comparison to the eutectic Sn:Bi
alloy with respect to the production of both carbon particles and hydrogen from the propane/butane mixture source. One may see the difference with respect to the particles trapped on the surfaces of the alloys in FIG.4 and FIG.5.

FIG. 4: Carbon on the surface of Sn:Bi alloy (app. 150x magnification)

FIG. 5: Carbon on the surface of Sn:Bi:Pb alloy (app. 150x magnification)

Partially this difference might be explained by the shaking of the Pb containing alloy during injection of the gas to a gas chromatograph. The injection was organized so, that the whole alloy in the test tube was moved by a pressure drop. The measured maximum and minimum contents of hydrogen in the gas were 158.7 ppm and 60 ppm respectively. Small amounts of intermediate products of propane/butane decomposition such as ethylene and propylene were also present in the gas. Also, the hydrogen content was approximately three
times higher in the experiments with an eutectic Sn:Bi alloy compared to the experiments with Sn:Bi:Pb alloy.

Because of trapping of carbon particles and hydrogen inside the alloy during its solidification, a slow pressure drop was observed in the tube between the test glass and a barrier volume containing water. Water from this volume was sucked into the alloy during 8 hours after cooling of all the parts in the setup to room temperature. This is due to a diffusion of hydrogen out from the porous structure in the solidified alloy. Furthermore, by its replacement with the propane/butane mixture resting in the tube. In the case of a tight tube connection between the test glass and a barrier volume with water, the water may come to a contact with the alloy due to a pressure drop in the tube.

B. Results of the experiments using the second experimental setup

Carbon particles are produced during the periodic blowing, when the pump is switched on and off. FIG.6 shows a photo of the filter before and after an experiment using setup No. 2 described in FIG.2. During the experiment the particles and propane/butane mixture were moving in a loop above the alloy. Also the alloy was under-cooled by this motion and melted when this motions was stopped.

The particles were also observed in the flexible intermediate volume, where agglomerations in the forms of triangles were found. These agglomerations were easily moved (they were jumping) by static electricity from fingers while touching the surface of a partially transparent volume.

The gas, which was collected in the intermediate volume along with the particles, was injected to the Agilent Micro GC in the end of an experiment. These measurements showed a maximum percentage for the hydrogen (0.1%) among the other hydrocarbons in mixture. Previous injections showed varying composition of the mixture, which was originated from the source propane/butane mixture: C3H8 - 16.78 %, iC4H10 - 18.22%, nC4H10 - 65.0 %. The most probable reasons for the variations are the periodic start-stop regime of the flow and pretty big volume of the gas in the loop. The results of four short-time characteristic series of measurements s are presented in the TABLE I.

The carbon particles were also deposited on the surface of a magnet, which was ap-
plied for mixing of chopped sticks of the alloy before it was melted. At the same time an appearance of the tiny black bars was noticed. They were mixed by a magnet together with the sticks in the glass bulb. When the alloy was melted, these bars disappeared in the melt. Their expected appearance took place during the experiment using setup No 3, which is shown in FIG.3.

C. Results of the experiments using the third experimental setup

The photo of the bars, which look like foils detached from the ends of the sticks during their collisional mixing, is presented in the FIG.7.

These bars contain carbon depositions, which are more extensive in comparison to the depositions on the fractal surfaces on the ends of the alloy sticks (see FIG.8-9). Because of this, the bars looked black under observation by a naked eye. Their lengths were comparable with the diameter of the sticks (see the FIG.10), but some of them could form the bars of a double length by electrostatic sticking one to another (see the FIG.7).
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**TABLE I: Gas measurements in the experiment using setup No 2**

![FIG. 7: Foils detached from the alloy sticks](image)

Measurements using energy dispersive spectroscopy (EDS) at the electron microscope showed the following percentage of the elements in the surface of a tiny bar near the end of an alloy stick in FIG.10: 37.82% C, 27.81 % Sn, 34.37% Bi. These are the weights in %. As the surface of the bar looks black, one may assume that a deposition of carbon particles on it is pretty dense. This is supported by the atomic ratios given by the EDS measurements: 88.76 % C, 6.60 % Sn, 4.64 % Bi. However, these values are not the same for every chosen bar with such a deposition. Some EDS measurements could also show a small presence of Fe on the bars. This element might appear on the bars from the cutters applied to cut the
alloy sticks.

In opposite to the results of the experiment using setup No 2, no visible depositions of the carbon particles appeared on the filter in the loop during approximately the same time of the experiments.

The same note applies for the experiments using setup No 3, where flexible volume
FIG. 10: Black bar (foil) near the end of chopped alloy stick

with the propane/butane mixture was replaced by the volume with biogas from Stockholm Vatten AB production facilities having 66.36 % methane and 33.63 % carbon dioxide. Even if percentage of the hydrocarbon in this mixture is not big, the noticeable amounts of hydrogen were measured during the experiments using continuous and start-stop blowing regimes of alloy application. In this experiment the temperature of molten alloy didn’t exceed 170°C. The results of gas measurements are presented in the Table II.

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TABLE II: Biogas measurements in the experiment using setup No 3
D. Discussion

The most valuable result of the experiments using pulsating flow of the gas is in the confirmation of a special role of the fractal surfaces participating in the process of decomposition of this gas.

The appearance of carbon particles in the case of interaction of hydrocarbons with mechanically created fractal surfaces being subjected to the pulsating blowing provides more support for the hypothesis, which was formulated by a correspondent author of this paper in [15]. This hypothesis explains the decrease of the activation energy of pyrolysis reactions in samples of wood and other biomasses having extensive internal gradients of temperature and pressure. The decrease of activation energy is analyzed in [15] from the point of view of a fundamental fractal theory [11–13]. Its explanation using the theory receives illustrative support in the experiments reported in this paper. Fractal surfaces of pores in the wood samples, which have extensive transport of the phase changing hydrocarbons moving in the pores due to the dynamic gradients of pressure and temperature, are analogous to the fractal surfaces of under-cooled Sn:Bi alloy. Furthermore, to the fractal surfaces on the ends of the chopped alloy sticks. The dynamic gradients of pressure and temperature with respect to the experimental conditions applied in the described experiments are created by the open volume of gas, by the wire heater, and by the heater beneath the test glass. Here the alloy and gas are driven by the membrane pump.

These conditions are necessary for a nontrivial Laplacian from the cited from the paper [12] formula (1) for the generalized quantum force $Q$ appearing for the system with the mass $m$ in the dynamic conditions of energy and mass transfer driven by the potential $P$:

$$
\frac{Q}{m} = -2D^2\nabla \left( \frac{\Delta \sqrt{P}}{\sqrt{P}} \right).
$$

(1)

Where the constant $D$ is assumed to be proportional to the coefficient in the equation describing the dynamic heat or mass transfer driven by the potential $P$. This transfer spatially includes the oscillator, which obtains an additional energy due to the presence of the transfer and due to the hypothetical fractality of space-time.

One may consider hydrogen-carbon bonds in molecules of hydrocarbons as approximate examples of the harmonic oscillators. If those oscillations are described by a standard equation $x = a \cdot \cos(\omega t)$, one can calculate the additional energy $(Dw)$ to be gained by such
oscillators accordingly to the formula (2) cited from [13]:

\[ E = Dw + \frac{a^2 w^2}{2}. \]  \hfill (2)

Where \( w \) is the frequency and \( a \) is the amplitude of oscillations.

The consideration in [13] was made for the case with the absence of an explicitly present fractal geometry, which may change the dynamics of the oscillator. To show the presence of this geometry in the geodesic motions of space-time, one needs to apply a special retro-active force loop to the oscillator. The purpose of this retro-active force loop is to tune the form of oscillations to the form predicted by the theory for oscillations of a generalized quantum wave packet.

According to our understanding of the basic concepts of the theory [11], such an artificial tuning is unnecessary in the case of natural fractal geometry being present along with the presence of dynamic potentials driving transfer processes involving oscillators of different sizes and scales. In such a case, the oscillators may indirectly obtain an additional energy for their oscillations due to the appearance of the generalized quantum potentials, which have origin at the presence of dynamic potential gradients, fractal properties of space-time, and fractal properties of experimental environments and conditions.

One may assume that if intermolecular vibrations in hydrocarbons will obtain more energy in such an indirect way while they pass along a fractal surface, the probability of a hydrocarbons’ decomposition due to a collisional energy exchange between their molecules will be increased. This might explain the appearance of carbon particles and their agglomerations after the passage of the propane/butane gas mixture via the filter in the loop.

From another point of view, one may explain the same process of decomposition by the mechanically assisted collisional energy input to molecules while their pressure driven passage in a proximity to the rough fractal surface. However, in such a case, one has to assume that the lowest scale of a metallic fractal is comparable to the scale of molecules of hydrocarbons. Even if that could be shown by means of microscopy, the effect of a collisional energy transfer to the tiny boundary layer of gas molecules, which may obtain additional energy input in a direct way due to the friction and convective heat transfer from the fractal surface, might be unnoticeable in respect to the thermal decomposition of the whole gas in the loop.

To estimate efficiency of such a direct mechanically assisted energy transfer to
molecules of the gas, both microscopical experimental research and molecular dynamic simulations seem to be necessary.

IV. CONCLUSIONS

As the focus of this paper is in determination of physical parameters, conditions and mechanisms of the low temperature pyrolysis of gaseous hydrocarbons taking place due to their motion along the fractal interfaces with the alloys, we may report that:

1. The experiments on a low temperature mechanically assisted pyrolysis of propane/butane mixture using several experimental setups showed a possibility to initiate the process at the gas temperatures starting from 70°C.

2. A mechanical assistance for the process was provided by the pulsating blowing of the gas, which moves along fractal surfaces that were created in the following two distinct ways:
   a) By an under-cooling of the alloys having melting temperatures of 138°C and 140°C.
   b) By a mechanical fracturing of the Sn:Bi alloy.

3. It was not possible to determine exact physical mechanism of the energy transfer from the mechanical energy of a pulsating blowing to the energy of hydrogen-carbon bonds vibrations, whose increase due to the passage of the gas along the fractal surfaces is presumable reason for the breakage of the bonds at extremely low temperatures from the thermodynamic point of view.

4. Two probable mechanisms of the energy transfer to vibrations of the bonds were discussed. The first hypothetical mechanism of the energy transfer to the bonds being considered as oscillators in the field of the dynamic gradients being considered in the frame of a fundamental fractal theory [12] was offered for a consideration by the corresponding author in the paper [15] in application to the high temperature pyrolysis of big wood samples. This hypothetical mechanism receives an experimental support in the experiments being described in this paper.

5. An alternative mechanism assumes a direct mechanically assisted collisional energy transfer to the bonds of hydrocarbons due to the friction and convective heat transfer from the fractal surfaces. The proof or rejection of this or the first mechanism requires molecular dynamic simulations supported by microscopic characterizations of the applied fractal
surfaces. Furthermore, more exact measurements of parameters such as the temperature, pressure and velocity of hydrocarbons during their motion along the surfaces are required. However, this is a topic of a future study.

V. ACKNOWLEDGMENTS

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