

Fe-Mo/Al₂O₃ catalyst for methane decomposition synthesized by solution combustion technique using oxalic acid

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ABSTRACT This work reports the synthesis of 80%Fe–10%Mo/10% Al₂O₃ catalysts via solution combustion synthesis using oxalic acid as fuel. The catalysts were tested in methane decomposition at 850 °C and 1 atm for 6 h. The synthesized samples were characterized by X-ray diffraction, transmission electron microscopy, photosedimentation analysis, etc. The specific carbon yield ranged from 5.8 to 8.4 g/gcat and the hydrogen yield from 1.0 to 1.4 mol/g_{cat}, respectively. Catalytic decomposition of methane over Fe–Mo/Al₂O₃ led to the formation of mixture of bamboo-like carbon nanotubes and multi-walled carbon nanotubes. It was found that the highest yield of MWCNTs was reached at fuel-to-oxidizer ratio of 0.022.

KEYWORDS catalysis; solution combustion; methane; catalyst; Fe-Mo-catalyst.

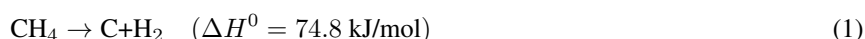
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1. Introduction

Currently, the development of technology for processing of associated petroleum gas (APG), natural gas and other gases containing C₁–C₄ hydrocarbons is a pressing issue for many oil-producing companies. Solving this problem will improve the environmental situation in Russia. A modern and economically efficient method for their processing is low-temperature pyrolysis, which occurs at temperatures up to 1000 °C. This process is simple to operate, easily scalable, and the reaction products are free of carbon oxides, making it a “CO_x-neutral” method. Furthermore, the process leads to the formation of hydrogen and nanostructured carbon. The production of the latter, as a product with specific added value, is considered an advantage from an economic perspective due to its wide range of applications [1]. Hydrogen can be used in metallurgy, hydrogen energy, petrochemicals, the chemical industry, etc. Carbon nanomaterials can be applied in environmental protection (as sorbents, in gas sensing) [2–5], as additives for composite materials [6–9], in electrochemical devices (supercapacitors, batteries) [10, 11], among other uses.

Low-temperature pyrolysis can be implemented over a wide range of temperatures (500–1000 °C) and pressures (1–10 atm) in the presence of catalysts containing metals such as Ni, Fe, Co, etc [12–16]. In this technology, the catalyst plays a primary role; its composition affects the process temperature, and its synthesis method impacts the catalyst production cost. The formation of hydrogen and carbon nanofibers is based on the decomposition reaction of the initial methane into carbon, hydrogen is passing according the reaction (1) [17–20].



The solution combustion synthesis (SCS) can be used for synthesis of catalysts for methane decomposition and related processes [21–24]. This method is relatively simple, rapid, and is a single-step process. It is based on a redox reaction between metal nitrates and reducing agents (so-called fuels), such as urotropine, glycine, oxalic acid, etc. [18].

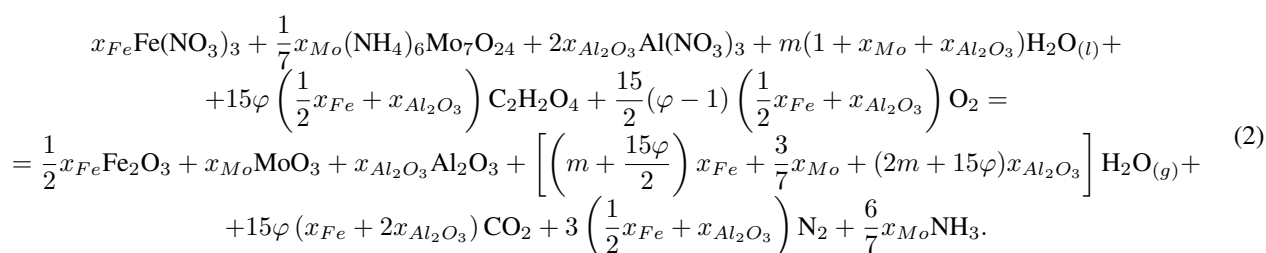
The oxalic acid-based SCS is applied for synthesis of Ni-containing catalysts [25, 26], CoCr_2O_4 ceramic pigment nanoparticles [27], hexagonal WO_3 [28], CuO [29] and other materials. However, the results of the use of oxalic acid for SCS of Fe-Mo/ Al_2O_3 for decomposition of methane and even for production of multi-walled carbon nanotubes (MWCNTs) are almost absent. The system is typically applied for synthesis of MWCNTs [30–32].

This work is devoted to investigation of the Fe-Mo/ Al_2O_3 catalysts using oxalic acid as a fuel for the methane decomposition reaction. The catalysts were tested and characterized at different fuel-to-oxidizer ratios. An analysis of the catalytic activity, crystallite size, phase composition, particle size of the resulting carbon nanomaterial was conducted.

2. Materials and methods

A series of 80%Fe–10%Mo/10% Al_2O_3 catalyst for the synthesis of MWCNTs was synthesized. Oxalic acid was used as the fuel in the catalyst systems under investigation. The implementation of catalyst synthesis via the SCS technique requires the calculations of fuel-to-oxidizer ratio φ . For this purpose, an approach based on thermodynamic calculations of the redox reaction is employed. The combustion reaction in an oxygen-containing environment, involving a metal nitrates, ammonium paramolybdate, and oxalic acid (fuel), can be derived by combining the decomposition reactions of metal nitrates to metal oxides in the presence of water, according to the reactions below.

Thus, the general combustion equation for the system under consideration can be obtained (Equation 2).



The investigation of the kinetic patterns of the process for producing carbon nanofibers and hydrogen via the catalytic decomposition of C1–C4 hydrocarbons was carried out using the catalyst samples described above. The testing was performed in a laboratory-scale quartz reactor at atmospheric pressure. The concentrations of the reagents and reaction products were measured using a Khromos GC-1000 gas chromatograph. A schematic diagram of the setup is shown in Fig. 1.

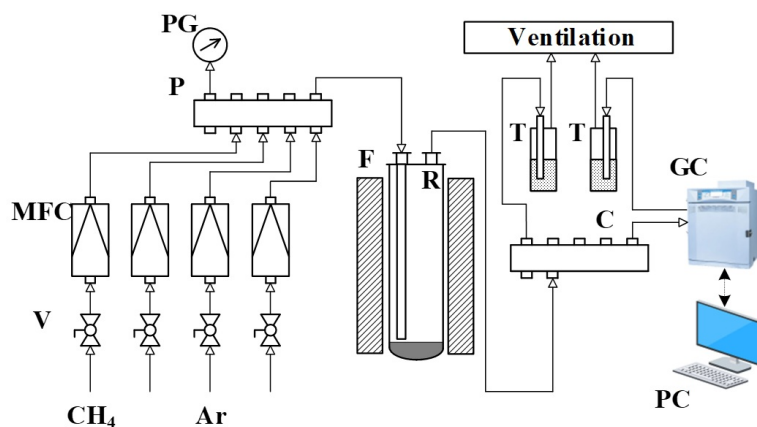


FIG. 1. Schematic diagram of the catalytic setup equipped with a quartz reactor: PG – Pressure Gauge; P – pipeline; MFC – Mass flow controller; V – Valve; F – Furnace (500–1000 °C); R – Reactor; T – Water trap; GC – Gas chromatograph; PC – Personal Computer

The quartz reactor is a vertical flask with an internal diameter of 28 mm and a height of 220 mm, which is hermetically sealed to a metal housing via a swage nut with a gasket made of thermally expanded graphite.

Due to the presence of iron and molybdenum oxides, the catalysts of the system 80 wt.% Fe – 10 wt.% Mo / 10 wt.% Al_2O_3 were pre-reduced in a hydrogen flow at 430 °C for 1 h. Although nickel-containing systems obtained by SCS can be initiated without reduction with hydrogen [33], the 80 wt.% Fe – 10 wt.% Mo / 10 wt.% Al_2O_3 system, which is oriented more towards the production of MWCNTs, becomes active only after reduction with H_2 . On one hand, this is a disadvantage for the industrial implementation of the process; on the other hand, it is typical behavior for Fe-containing systems [34,35]. The operating temperature was 850 °C. The use of higher temperatures is not advisable from an economic point of view, as the process must be conducted with minimal costs, which is important for its piloting and semi-industrial testing. To accelerate the testing, the typical duration of the catalytic experiment was set at 6 h.

Transmission electron microscopy (TEM) was performed using a JEOL JEM-2200 FS-CS instrument with a resolution of up to 0.1 nm. The accelerating voltage was 80–200 kV.

3. Results and Discussion

The considered 80 wt.% Fe – 10 wt.% Mo/10 wt.% Al₂O₃ catalysts exhibited catalytic activity only after a preliminary reduction stage in a hydrogen flow at 430 °C (for 1 h). The hydrogen concentration value reaches its maximum level at the initial moment, ranging from approximately 11–20 vol.% (Fig. 2a), which is explained by the completion of the catalyst reduction stage and the initiation of its operation. Over time, the hydrogen concentration in the reaction medium gradually decreases, reaching values of approximately 8–14 vol.% (Fig. 2b). Subsequently, the catalyst deactivation rate decreases significantly, and a smooth decline is observed over 4 h, with the hydrogen concentration stabilizing at a level of approximately 4–6 vol.%. Varying the fuel excess had an insignificant effect on the catalyst's activity. It can be noted that compared to other common fuels (e.g., urea, glycine), the use of oxalic acid leads to a higher adiabatic combustion temperature during solution combustion synthesis due to its more negative enthalpy of formation. However, oxalic acid generates the smallest amount of gaseous products upon combustion, which in turn affects the porosity and specific surface area of the resulting catalysts.

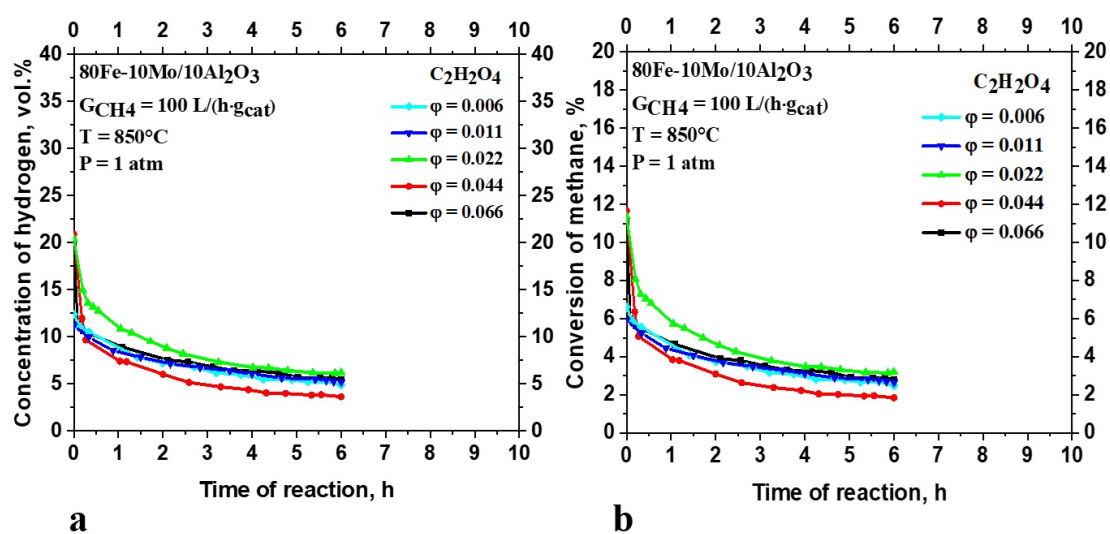


FIG. 2. (a) Hydrogen concentration and (b) methane conversion versus reaction time for the 80 wt.% Fe – 10 wt.% Mo / 10 wt.% Al₂O₃ catalyst (fuel: oxalic acid; LHSV = 8.9 h⁻¹)

Table 1 presents the results of the catalytic tests for the catalysts in the laboratory quartz reactor for the methane decomposition reaction.

TABLE 1. The results of catalytic tests

No	φ	Catalytic tests		
		t, h	$Y_C, g/g_{cat}$	$Y_{H_2}, mol/g_{cat}$
1	0.006	6	6.1	1.0
2	0.011		5.8	1.0
3	0.022		8.4	1.4
4	0.044		5.8	1.0
5	0.066		6.9	1.1

A clear optimum of catalytic activity is observed at $\varphi = 0.022$. At this value, the yields of carbon and hydrogen are maximum (8.4 g/g_{cat} and 1.4 mol/g_{cat}). This indicates that it is with this rather low fuel ratio that the most favorable conditions are created for the formation of a highly active catalytic phase. The highest rate of carbon formation was 1.4 g/(g_{cat}*h).

TEM images (Fig. 3a-c) of the catalysts represented the aggregated structures, which significantly different from the one obtained by sol-gel, impregnation and other techniques [36–39]. This is caused by high temperature reached during

combustion and the smelted-like particle morphology is typical for SCS-based powders. The diameter of nanoparticles ranged within 5–10 nm.

The Fe–Mo/ Al_2O_3 catalyst at 850 °C forms classical structures characteristic of multi-walled carbon nanotubes (MWCNTs) and bamboo-like carbon nanotubes (Fig. 3d-f).

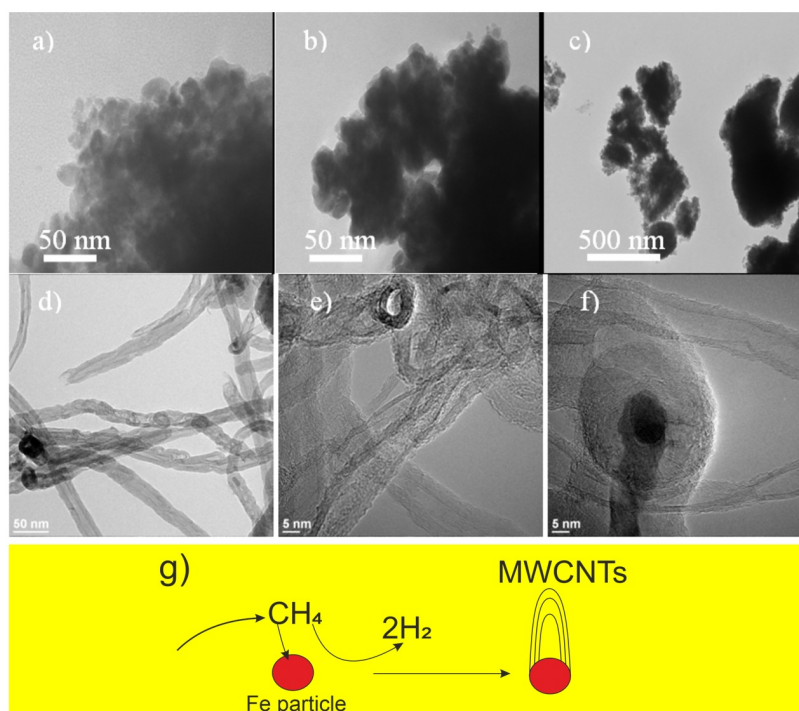


FIG. 3. TEM images of the (a-c) catalyst ($\varphi = 0.022$) and (d-f) carbon nanomaterial grown over 80 % Fe – 10 % Mo/ Al_2O_3 catalyst (850 °C, 1 atm), mechanism of methane decomposition and MWCNTs growing (g)

This suggests that tip-growth mechanism operate simultaneously during methane decomposition over the Fe–Mo/ Al_2O_3 catalyst (Fig. 3g) [40,41].

According to X-ray diffraction patterns, the catalysts of the 80 wt.% Fe – 10 wt.% Mo / 10 wt.% Al_2O_3 system synthesized with oxalic acid consisted fully of the $\text{Fe}_2(\text{MoO}_4)_3$ phase (JCPDS 31-642, monoclinic) (Fig. 4).

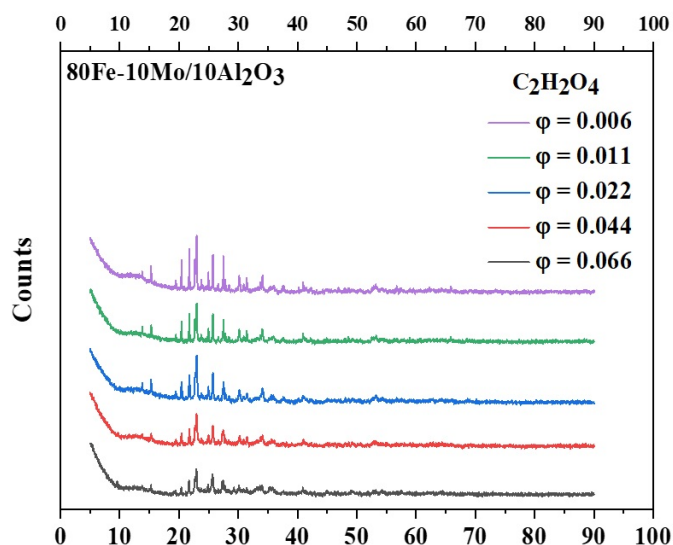


FIG. 4. X-ray diffraction patterns of the synthesized 80 wt.% Fe – 10 wt.% Mo / 10 wt.% Al_2O_3 catalysts using oxalic acid (Cu $K\alpha$ radiation, 1.54 Å)

It was found that the phase composition depends on the fuel-to-oxidizer ratio. Reflections related to the fuel are almost entirely absent, although elemental analysis of virtually all catalysts obtained by combustion synthesis typically

indicates residues of the fuel or compounds formed during its transformation (ranges within 2–4 wt.% according to energy-dispersive X-ray spectroscopy). This latter aspect has been investigated very little, and such studies represent a subject for separate research due to the complexity of the task, the vast number of compounds that fuels can form with elements in the initial mixture, and especially in the case of bi- and trimetallic systems, where the range of such compounds (and the problem of their identification) increases significantly.

For Fe₂(MoO₄)₃ the crystallite size generally increases with increasing φ (from 83.2 nm at $\varphi = 0.066$ to more than 100 nm (the large grains is not possible to determine due to high error) at $\varphi = 0.022$ –0.044). The exception is the sample with $\varphi = 0.066$, where the growth in φ induced the lower crystallite size. It is hypothesized that the most active catalyst ($\varphi = 0.022$) has a relatively high crystallite size. Therefore, it can be supposed, that this value does not bring a linear effect on the operation of the catalyst. Probably, the catalysts with fine grains operated at higher methane conversion during first minutes of process and the majority of active sites blocked during initial period. Therefore, usually, the catalysts with high crystallite size possessed higher time until reaching deactivation and higher yield of carbon and hydrogen. The relatively low crystallite size at $\varphi = 0.006$ –0.011 indicated the low-temperature synthesis and the growth in fuel-to-oxidizer ratio led to sharp growth in crystallite size caused by fuel-driven temperature increase. Further growth ($\varphi > 0.044$) induced the decrease in crystallite size, but the change in lattice parameters compared to other samples (Table 2). Even when the lattice of sample $\varphi = 0.066$ under stress state, the activity of such catalyst is not so high.

Typically, the particle size of the catalysts is large enough and somehow correlates with the compounds synthesized by SCS, representing the smelted powder of micron size particles [42–45]. It is important to note that these data represent catalyst agglomerates, not the primary particles of the active Fe-Mo phase. The analysis of $\varphi = 0.22$ sample was carried out. The photo-sedimentation analysis reveals a broad particle size spectrum ranging from 1.00 to 120.0 μm . The key statistical parameters indicate a median diameter (d_{50}) of 65.1 μm . The distribution is multimodal, with distinct peaks identified at 2.8, 10.2, 18.2, 50.3, and 67.2 μm . The granular composition is characterized by a high content of particles larger than 10.0 μm , constituting 93.4% of the sample, while only 11.5% of particles are finer than 14.0 μm . Compliance with technical specifications was confirmed by the characteristic percentiles: $d_3 = 116.8 \mu\text{m}$, $d_{50} = 65.1 \mu\text{m}$, and $d_{94} = 9.7 \mu\text{m}$. In summary, the catalyst is a polydisperse powder with a predominant fraction in the medium size range around 65 μm and a non-uniform, multimodal distribution, which is a typical characteristic for materials synthesized by the solution combustion method.

TABLE 2. Results of the X-ray diffraction of the synthesized catalyst samples

φ	Crystallite size of Fe ₂ (MoO ₄) ₃ phase, nm	Lattice parameters, Å		
		a	b	c
0.006	83.2±5	15.6828±0.004	9.2278±0.002	18.2146±0.004
0.011	66.3±5.2	15.6826±0.008	9.2296±0.002	18.2121±0.008
0.022	> 100	15.6850±0.004	9.2302±0.0012	18.2152±0.003
0.044	> 100	15.6863±0.0012	9.2303±0.0008	18.2157±0.0016
0.066	60.4±6.6	15.6968±0.014	9.2375±0.006	18.2245±0.012

TABLE 3. Comparison of yield of CNTs obtained over various catalysts

Reference	Catalyst	Gas (feedstock)	Carbon yield (time on stream), g/g _{cat}
[46]	50%Fe-50%Co (non-supported)	CH ₄	0.6 g/g _{cat} (7 h)
[47]	40%(Fe ₂ Mo)/Al ₂ O ₃	C ₂ H ₄	7.7 g/g _{cat} (n/a)
[48]	FeMo/MgO	CH ₄	5.77 g/g _{cat} (n/a)
[49]	Fe-Co/TiO ₂	C ₂ H ₂	3.4 g/g _{cat} (n/a)
This work	80%Fe-10%Mo/Al ₂ O ₃	CH ₄	8.4 g/g _{cat} (6 h)

Although the crystallite size of Fe₂(MoO₄)₃ varied significantly with φ (from 60 to more than 100 nm), no direct correlation with the catalytic activity was observed. This can be attributed to several factors. First, the Fe₂(MoO₄)₃ phase is reduced to metallic Fe(Mo) nanoparticles under H₂ at 430 °C, and the size of these active particles does not necessarily reflect the initial crystallite dimensions. Second, the Al₂O₃ formed during combustion synthesis, acts as a

structural promoter that inhibits sintering of the metallic phase, thereby stabilizing a wide range of particle sizes. Third, the presence of Mo enhances the metal–support interaction and may alter the growth mechanism of carbon nanotubes. Finally, the catalyst particles are polydisperse (1–120 μm) with complex internal porosity, so the accessibility of active sites is governed by textural properties rather than the primary crystallite size. The highest activity observed at $\varphi = 0.022$ is therefore attributed to an optimal unit cell parameters and high crystallite size of $\text{Fe}_2(\text{MoO}_4)_3$ phase.

For comparison, similar works on Fe-containing catalysts were analyzed and presented in Table 3 and showing the efficiency of the catalyst under study. However, direct comparison of carbon yields in Table 3 should be made with caution, as the data originate from different feedstocks (CH_4 , C_2H_4 , C_2H_2) and reaction conditions (temperature, pressure, time on stream), which significantly affect catalyst performance.

4. Conclusions

The SCS method with oxalic acid as a fuel has proven its effectiveness for obtaining Fe–Mo/ Al_2O_3 catalysts for the decomposition of methane. The catalysts showed activity and produced bamboo-like carbon nanotubes and hydrogen. The fuel-to-oxidizer ratio (φ) is a critically important parameter that determines the phase composition, the size of crystallites and, as a result, the catalytic activity. It was found that there is an optimal value of $\varphi = 0.022$. The catalytic activity (carbon yield of 8.4 g/g_{cat}) depends non-linearly on φ . Fe-based catalysts require a longer pre-reduction stage, which is a disadvantage for industrial applications compared to some Ni-catalysts. There is also a gradual deactivation during the reaction. The results obtained are of great value for optimizing the synthesis of catalysts by the SCS method.

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