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Stacked-cup multiwall carbon nanotubes as components of energy-intensive suspension jet fuels

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Objectives. *The addition of high-density carbon materials to jet fuels can lead to a significant increase in the volumetric energy of the fuel combustion. The purpose of the current study was to thermodynamically analyze the possibility of obtaining model hydrocarbon fuels from toluene and T-1 using stacked-cup multiwall carbon nanotubes (MWCNTs).*

Methods. *Bomb combustion calorimetry was used to define the combustion energy of the MWCNTs in the crystalline state. The temperature dependence of the MWCNTs' heat capacity in the range 5–370 K and the fusion parameters were estimated using low-temperature adiabatic calorimetry. The physical density of MWCNTs was measured using the pycnometric method. The sedimentation stability of the mixtures of MWCNTs with liquids was determined using centrifugation at 7000 g. The calculations were carried out in MS Excel.*

Results. *The energy and enthalpy of combustion of a technical sample of MWCNTs in the crystalline state were determined. Based on the smoothed heat capacity values, the standard thermodynamic functions (enthalpy, entropy, and Gibbs reduced energy) of MWCNTs in the crystalline state were obtained in a temperature range of 0–2000 K. The extrapolation of the MWCNTs' heat capacity was carried out at a temperature of up to 2000 K using the heat capacity of crystalline graphite. It has been established that mixtures of MWCNTs with liquids containing more than 33 mass % of MWCNTs are stable during centrifugal sedimentation at 7000 g. For the toluene–MWCNTs and fuel T-1–MWCNTs model systems, the specific and volumetric combustion energies, the adiabatic combustion temperatures, and the conditional final maximum speed of the model rockets with fuel of various compositions were also calculated.*

Conclusions. *The thermodynamic analysis showed that the addition of MWCNTs can significantly increase the volumetric energy intensity of traditional jet fuels, which can in turn improve the operational characteristics of drones and rockets.*

Keywords: *model hydrocarbon fuel, energy intensity, cup multiwall carbon nanotubes, specific energy of combustion, volumetric energy of combustion, adiabatic combustion temperature, specific impulse, conditional final maximum speed, industrial implementation.*

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Многослойные углеродные нанотрубки – компонент энергоемких суспензионных реактивных горючих

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Цели. *Добавление высокоплотных углеродных материалов в реактивные топлива может привести к значительному увеличению его объемной энергоемкости. Цель работы заключалась в проведении термодинамического анализа возможности получения модельных углеводородных топлив из толуола и Т-1 с многослойными углеродными нанотрубками (МУНТ).*
Методы. *Свойства МУНТ были исследованы при помощи следующих методов: энергия сгорания в кристаллическом состоянии определена методом бомбовой калориметрии, температурная зависимость теплоемкости в интервале 5–370 K – методом адиабатической калориметрии, физическая плотность – пикнометрическим методом, седиментационная устойчивость смесей с жидкостями – центрифугированием при 7000 g. Расчеты проводились в программе MS Excel.*
Результаты. *Определены энергия и энтальпия сгорания технического образца МУНТ в кристаллическом состоянии. На основании сглаженных значений теплоемкости получены стандартные термодинамические функции (энтальпия, энтропия и приведенная энергия Гиббса) МУНТ в кристаллическом состоянии в интервале 0–2000 K. Экстраполяция теплоемкости МУНТ до температуры 2000 K проведена с использованием теплоемкости кристаллического графита. Установлено, что смеси МУНТ с жидкостями, содержащими МУНТ более 33 масс. %, седиментационно устойчивы в центрифуге при 7000 g. Для модельных систем толуол–МУНТ, горючее Т-1–МУНТ вычислены массовые и объемные энергии сгорания, адиабатические температуры горения, условная конечная максимальная скорость модельных ракет с горючим различных составов.*
Выводы. *Термодинамический анализ показал, что добавление МУНТ существенно повышает объемную энергоемкость традиционных реактивных топлив, что должно приводить к улучшению эксплуатационных характеристик летательных аппаратов.*

Ключевые слова: *модельное углеводородное топливо, энергоемкость, многослойные углеродные нанотрубки, массовая теплота сгорания, объемная теплота сгорания, адиабатическая температура горения, удельный импульс, условная конечная максимальная скорость, промышленное внедрение.*

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INTRODUCTION

The development of energy-intensive chemical fuels for unmanned aerial vehicles and various jet engines is undoubtedly an urgent task. Carbon materials, such as carbon black and C_{60} fullerite, are often used as components of energy-intensive liquid (pasty) and solid (reactive) fuels due to their high physical density, high combustion energy, and the formation of non-condensable products (CO_2) during combustion [1].

This paper presents the results of a study on the thermodynamic properties and density of a technical sample of multiwall carbon nanotubes (MWCNTs, *Vision Development*, Japan). Some of the commercial physicochemical and structural parameters of the investigated MWCNTs are outlined in Table 1.

Table 1. Physicochemical parameters of MWCNTs as provided by the supplier

Parameter	Value
Physical state	Black powder
Average length, m	$\leq(1–2) \times 10^{-6}$
Average inner diameter, m	$\sim 50 \times 10^{-9}$
Outer diameter, m	$(10–80) \times 10^{-9}$
Thermal stability, K	<973

Kabo *et al.* [2, 3] have demonstrated that the density and combustion energy of MWCNTs in terms of 100% carbon content are almost similar to the corresponding parameters of crystalline graphite (Table 2). Moreover, the physical density of MWCNTs is significantly higher than the density of carbon black and fullerite C_{60} (by 16 and 32%, respectively), indicating them as a promising component for the development of energy-intensive fuels in mixtures with hydrocarbons. Fullerite C_{60} has a higher (~ by 10%) specific energy of combustion [4, 5] than graphite and MWCNTs, whereas its physical density is significantly lower than that of graphite by about 30% (Table 2). Fullerite C_{60} is also slightly soluble in hydrocarbons, meaning that the internal cavity of the C_{60} molecules is not filled with liquids, thus presenting a low potential as a component of liquid fuels.

Given our earlier report that liquids with low molecular weight can fill the inner cavity of MWCNTs, while the sorption enthalpy of the liquid components (ionic liquids) is very small [7], this paper presents estimates of the energy efficiency of combustible toluene–MWCNTs and T-1–MWCNTs model systems.

MATERIALS AND METHODS

The density of the MWCNTs was measured in a glass pycnometer with a volume of 0.8876 ± 0.0009 cm^3 using bidistilled water (ρ = 998.3 $kg·m^{-3}$) (292 K) [3] and freshly distilled toluene (ρ = 867.8 $kg·m^{-3}$) [2] as pycnometric liquids. A pycnometer filled with nanotubes and a pycnometric fluid was heated in boiling water and, after cooling to ~293 K without contact with air, was filled to the mark with a pycnometric fluid and weighed. The average physical density of MWCNTs was 2210 ± 22 $kg·m^{-3}$ [2], which practically corresponds to the density of hexagonal graphite 2265 $kg·m^{-3}$ [6]. Moreover, the bulk density of MWCNTs has been reported to be approximately 400 $kg·m^{-3}$ [2].

The combustion enthalpy of a technical sample of MWCNTs was determined in an automated combustion calorimeter [8] with a 326 cm^3 calorimetric bomb and an energy equivalent of 14605 ± 3 $J·K^{-1}$. The MWCNTs samples were dried at 130 and 200°C under vacuum ($P \approx 100$ Pa). According to the results of burning in a calorimetric bomb, the ash content in the MWCNTs was estimated to be 0.88 ± 0.07%, which corresponds to 0.69% of nickel metal used as a synthetic catalyst. It was also shown that metallic nickel cannot be removed from the MWCNTs neither by magnetic separation nor by dissolution in nitric acid within 24 h. According to the results that were obtained after burning the vacuum-dried technical samples of MWCNTs in plastic bags with cotton thread for ignition, the average combustion energy was determined as $\Delta_c U°(298.15\ K) = -(32336 \pm 13)\ kJ·kg^{-1}$ [2].

The heat capacity of a technical sample of MWCNTs in the range of 5–370 K was measured in a TAU-10 adiabatic calorimeter [8]. It has also been shown in [2, 3] that the heat capacity of MWCNTs, graphite, and fullerites C_{60}, C_{70} in a temperature range of 300–350 K differs only by 3%. The extrapolation of the specific heat C_p of the technical sample $MWCNT_{tech}$ from 400 to 2000 K was carried out by the equation:

$$C_p(MWCNT_{tech.}, T(K)) = 0.9931 C_p(graphite, T(K)) + 0.0069 C_p(Ni(cat.)) \times T(K) \quad (1)$$

The thermodynamic properties of the technical sample of MWCNTs in the range 0–2000 K are presented in Table 3.

RESULTS AND DISCUSSION

Physical and chemical properties of the MWCNTs–liquids (ionic and nonionic) mixtures

In a detailed study of the interaction of ionic and nonionic liquids with MWCNTs [7], it was found that nonionic liquids (toluene and water) penetrate the

Table 2. Combustion energy $-\Delta_c U°(298.15\ K)$ and density ρ of crystalline graphite, MWCNTs, and fullerite C_{60}

Property	Crystalline graphite	MWCNTs	C_{60}
$-\Delta_c U°(298.15\ K)$, $kJ·kg^{-1}$	32764 ± 4[1]	32336 ± 13 [2]	35802 [4]
ρ (298.15 K), $kg·m^{-3}$	2265 [6]	2210 ± 22 [2]	1720 [4]

[1] NIST Chemistry Webbook, SRD 69. Available from: webbook.nist.gov/chemistry/ [Accessed March 15, 2019]. https://doi.org/10.18434/T4D303

inner cavity of the MWCNTs when mixed at 293 K and P = 1 bar. In comparison, ionic liquids (IL) do not penetrate into the internal cavity of the MWCNTs at atmospheric pressure, but they completely penetrate into a MWCNT under vacuum conditions, thus allowing the determination of the cavity volume. Moreover, the enthalpy of mixing MWCNTs with ILs (e.g., 1-butyl-3-methylimidazolium hexafluorophosphate) does not exceed the sensitivity of the mixing calorimeter [7, 8]. Finally, it was demonstrated that the sedimentation stability of the MWCNT–IL systems during centrifugation at 7000 g depends on the components' ratio. In particular, at a mass content of MWCNTs ≥ 0.33 in the mixture, no separation of the systems was observed, implying that systems containing MWCNTs that exceed the bulk density are stable in sedimentation. Furthermore, based on computer simulations [9], it was established that the packing density (φ) of figures of different geometry depends slightly on their configuration and has a mean value of 0.58, which allows the estimation of the formation of the T-1–MWCNTs and toluene–MWCNTs compositions with a mass fraction of up to ω = 0.75.

Certain technical characteristics of the suspension fuel type C_nH_m–MWCNTs

The physicochemical and thermodynamic properties of the suspension fuel type C_nH_m–MWCNTs can be calculated based on the additivity of the corresponding compositions (Tables 4–6).

Table 3. Thermodynamic functions (standard heat capacities, entropies, enthalpies and Gibbs reduced energy) of MWCNTs (99.31 mass % C, 0.69 mass % Ni)

T*, K	C_p^o, J·kg^{-1}·K^{-1}	$\Delta_0^T S^o$, J·kg^{-1}·K^{-1}	$\Delta_0^T H^o$, kJ·kg^{-1}	Φ^o, J·kg^{-1}·K^{-1}
0	0	0	0	0
50	51.91	28.62	0.9388	9.846
100	158.5	95.64	6.082	34.82
150	290.2	184.0	17.20	69.32
200	437.8	287.5	35.36	110.7
250	591.6	401.7	61.10	157.3
298.15	733.7	518.1	93.04	206.1
300	739.0	522.7	94.40	208.0
370	932.0	697.5	153.0	284.0
400*	932.0	697.5	153.0	284.0
500	1261	1027	296.3	434.3
600	1453	1274	432.3	553.9
700	1598	1510	585.1	673.8
800	1710	1731	750.6	792.4
900	1796	1937	926.0	908.3
1000	1864	2130	1109	1021
1100	1921	2311	1298	1130
1200	1961	2480	1492	1236
1300	2009	2639	1691	1338
1400	2051	2789	1894	1436
1500	2092	2932	2101	1531
1600	2113	3068	2311	1623
1700	2134	3197	2524	1712
1800	2900	3340	2775	1798
1900	3004	3499	3070	1883
2000	2175	3633	3329	1968

*The thermodynamic properties of MWCNTs at 400–2000 K were extrapolated according to equation (1).

Table 4. Physicochemical properties of the fuel components

Component	Composition	M, g·mol^{-1}	ρ, kg·m^{-3}	$-\Delta_c H°(298.15)^*$, kJ·kg^{-1}	$-\Delta_c H°(298.15)^*$, MJ·m^{-3}
MWCNTs [2]	$CNi_{0.0014}$	12.094	2210	32336	71463
Toluene [1]	C_7H_8	92.141	867.1	40963	35519
Combustible T-1 [1]	$C_{12.8}H_{24.12}$	178.05	800	42900	34320
Carbon black [1]	C	12.011	1900	32781	62284

* Net calorific value.

Table 5. Lower enthalpies of combustion for the toluene–MWCNTs mixtures in stoichiometric quantities of oxygen and air at 298.15 K and P = 1 bar

ω(MWCNTs), mass fract.	Combustible combustion enthalpy		Fuel combustion enthalpy		
			with oxygen		with air
	$-\Delta_c H°$, MJ·kg^{-1}	$-\Delta_c H°$, MJ·dm^{-3}	$-\Delta_c H°$, MJ·kg^{-1}	$\Delta_c H°^*$, MJ·dm^{-3}	$-\Delta_c H°$, MJ·kg^{-1}
0	40.96	35.52	9.929	10.52	2.825
0.10	40.10	37.02	9.834	10.60	2.805
0.20	39.24	38.73	9.737	10.69	2.785
0.30	38.37	40.69	9.637	10.78	2.764
0.40	37.51	42.97	9.535	10.88	2.743
0.50	36.65	45.65	9.430	10.99	2.721
0.75	34.49	54.95	9.156	11.29	2.663

*When calculating the fuel density, the liquid oxygen density was considered to be $\rho(O_2)$ = 1140 kg·m^{-3} [1].

Table 6. Lower enthalpies of combustion for the T-1–MWCNTs mixtures in stoichiometric quantities of oxygen and air at 298.15 K and P = 1 bar

ω(MWCNTs), mass fract.	Combustible combustion enthalpy		Fuel combustion enthalpy		
			with oxygen		with air
	$-\Delta_c H°$, MJ·kg^{-1}	$-\Delta_c H°$, MJ·dm^{-3}	$-\Delta_c H°$, MJ·kg^{-1}	$\Delta_c H°^*$, MJ·dm^{-3}	$-\Delta_c H°$, MJ·kg^{-1}
0	42.90	34.32	9.786	10.17	2.747
10	41.84	35.76	9.708	10.27	2.735
20	40.79	37.40	9.627	10.38	2.722
30	39.73	39.31	9.544	10.50	2.710
40	38.67	41.54	9.457	10.62	2.696
50	37.62	44.19	9.367	10.76	2.682
75	34.98	53.66	9.128	11.15	2.643

*When calculating the fuel density, the liquid oxygen density was considered to be $\rho(O_2)$ = 1140 kg·m^{-3} [1].

In contrast, the calculation of the density of the mixtures of liquid fuels and MWCNTs does not obey the rules of additivity. Therefore, the density was determined as the ratio of the mass of the mixture to the sum of the volumes of the components included in the mixture, taking into account the composition.

The conventional adiabatic combustion temperatures (T_{ad}) were determined using the heat capacities of gaseous CO_2, H_2O, N_2, and Ar,[2,3] as follows:

$$-\Delta_c H_{298.15} = \int_{298.15}^{T_{ad}} a_i C_{p,i} dT, \quad (2)$$

where $\Delta H_{298.15}$ is the net calorific value of the fuel in air, a_i represents the mole fractions of the components of the fuel gases in the mixture, and $C_{p,i}$ is the heat capacity of the components in $J \cdot mol^{-1} \cdot K^{-1}$. The fuel gases consisted of CO_2, H_2O, N_2, and Ar, provided that there was no dissociation and conversion of combustion products. The composition of the air components was obtained from the State Standard[4] and the calculation results are presented in Table 7.

To assess the efficiency of the fuels including MWCNTs, the final gas flow rate (conditional specific impulse ($m \cdot s^{-1}$)) was calculated [10, 11] based on the following formula (3),

$$I_{spec} = W = \sqrt{2(-\Delta_c H) n_t}, \ m \cdot s^{-1}, \quad (3)$$

where $\Delta_c H$ is the lower enthalpy of fuel combustion with a stoichiometric amount of oxidizing agent ($J \cdot kg^{-1}$), and n_t is the thermal effective efficiency rocket engine determined by the ratio:

Table 7. Physicochemical characteristics of the combustible suspensions' toluene*–MWCNTs and T-1**–MWCNTs

Property	Mass fraction ω(MWCNTs) in fuel					
	0	0.20	0.30	0.40	0.50	0.75
toluene*–MWCNTs						
ρ, $kg \cdot m^{-3}$	867.1	987.1	1060	1146	1246	1593
T_{ad}, K (in O_2)	6227	6302	6343	6387	6433	6565
T_{ad}, K (in the air)	2507	2496	2491	2486	2480	2465
I_{spec}, $m \cdot s^{-1}$	2004	1990	1983	1975	1967	1946
P_{spec}, s	204.4	202.9	202.2	201.4	200.6	198.4
V_{max}, $m \cdot s^{-1}$	4615	4816	4928	5049	5181	5574
T-1–MWCNTs**						
ρ, $kg \cdot m^{-3}$	800	917	989	1074	1175	1534
T_{ad}, K (in O_2)	5738	5870	5944	6025	6114	6378
T_{ad}, K (in the air)	2408	2413	2417	2421	2425	2436
I_{spec}, $m \cdot s^{-1}$	1977	1969	1984	1959	1954	1940
P_{spec}, s	201.6	200.7	202.3	199.7	199.2	197.8
V_{max}, $m \cdot s^{-1}$	4553	4776	4950	5037	5186	5634

*toluene: C_7H_8; $M = 92.1$ $g \cdot mol^{-1}$; $C°_{298.15} = 1693$ $J \cdot kg^{-1} \cdot K^{-1}$ [1].
**combustible T-1: $C_{12.8}H_{24.12}$; $M = 178.1$ $g \cdot mol^{-1}$; $C°_{298.15} = 1921$ $J \cdot kg^{-1} \cdot K^{-1}$ [1].

[2] NIST Chemistry Webbook, SRD 69. Available from: webbook.nist.gov/chemistry/ [Accessed March 18, 2019] https://doi.org/10.18434/T4D303
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$$n_t = 1 - \left(\frac{P_0}{P}\right)^{\frac{k-1}{k}}, \quad (4)$$

where P and P_0 represent the pressure in the combustion chamber and at the nozzle exit, respectively (it is assumed that $P_0/P = 0.01$), and k is the adiabatic exponent, which depends on the molecular masses of the combustion products ($k = C_p/C_v$), provided that $k(T)$ = const. For mixtures of combustion products, the heat capacity was calculated as $C_p = \Sigma a_i C_{p,i}$, where a_i is the mole fraction of the component in the mixture and $C_{p,i}$ is the heat capacity of the component. Then, in the ideal gas approximation, $C_v = C_p - R$ applies.

Moreover, the specific thrust could be calculated by the following formula:

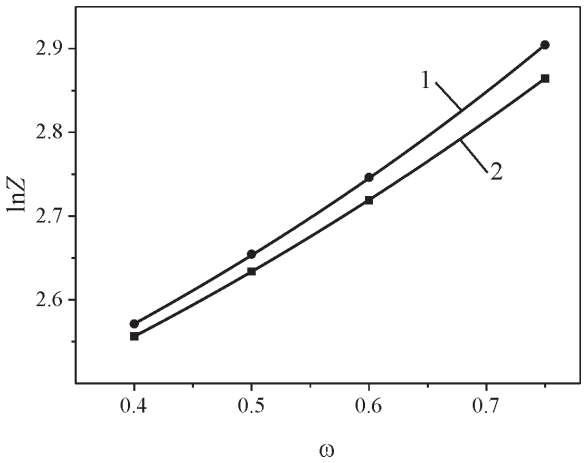
$$P_{spec} = \frac{I_{spec}}{g}, \text{ s}, \quad (5)$$

where g is the acceleration of gravity.

The conditional final (maximum) speed of the aircraft ($m \cdot s^{-1}$) at the time of the combustion completion without taking into account the gravity and aerodynamic drag was calculated by the Tsiolkovsky ratio:

$$V_{max} = I_{spec} \ln Z, \quad (6)$$

где $Z = 1 + \frac{m_{fuel}}{m_{str}}$; m_{fuel} is the mass of the fuel (kg), and m_{str} is the mass of the structure without fuel (kg). The parameter ln$Z$ was defined for a model rocket with mass ($m_{fuel} + m_{str}$) = 1000 kg without MWCNTs (ω = 0) and a constant m_{str} of 100 kg. The volume of the fuel tank (V_t) in this model was $900/\rho_{fuel}$. Accordingly,



Dependence of the parameter lnZ on the mass fraction of nanotubes:
1) for the mixture T-1–MWCNTs,
2) for the mixture toluene–MWCNTs.

the volume for toluene $V_т'$ was 1.0379 m^3 and that for fuel T-1 $V_т''$ = 1.125 m^3. The dependence of the parameter ln$Z$ on the mass fraction of nanotubes is shown in the following figure.

CONCLUSIONS

The calculations indicated that for fuels with MWCNTs, the conditional maximum speed of the model system increases by 24–26% at ω = 0.75, which will probably lead to a corresponding increase in the flight range of the aircraft. The report that catalysts based on carbon nanotubes with metal and oxide nanoparticles can control the burning rate [12], increases the potential of carbon nanotubes as components of energy-intensive fuels and arouses interest in such systems. However, their practical application requires further research, while additional technically important properties, such as viscosity, burning rate, etc., need to be investigated as well.

The authors declare no conflicts of interest.

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