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Research Paper

Fouling formed on SS316L tube surface from thermal oxidative degradation of *exo*-tetrahydrodicyclopentadiene

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HIGHLIGHTS

- The fouling of JP10 in autoxidative and intermediate regime was studied.
- The amount of the coke deposits of JP10 increases with the wall temperature.
- The deposits of JP10 were mainly in the form of amorphous carbon.

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ABSTRACT

Exo-tetrahydrodicyclopentadien called JP-10 is an endothermic hydrocarbon fuel with high-energy density being used in aircrafts. We have investigated the fouling of JP-10 on the inner surface of SS316L tube at wall temperature from 260 °C to 350 °C and at 3.84 MPa with a flow rate of 0.5 ml/min for 24 h. The fouling of JP-10 was influenced by wall temperature, fuel temperature and the metal surface. The average values of coke deposits were measured to be between 7.52 μg/cm^2 and 10.67 μg/cm^2 by carbon burn-off method, and the maximum values of local coke deposits and average values of coke deposits increase with the wall temperature. At 350 °C and 300 °C, the locations of peak deposition shifted upstream as the wall temperature increased. The nature and morphology of deposits were characterized by field emission scanning electron microscopy (FESEM) and temperature programmed oxidation (TPO) profiles, and the deposits of JP-10 were mainly in the form of amorphous carbon. Structurally more ordered deposits were also found, and it should be caused by the catalysis of iron and nickel in metal surface.

1. Introduction

The fouling or coking will be formed in fuel system of aircraft at high temperature when the fuel absorbs waste heat from aircraft component. The deposits will reduce the performance of heat exchangers and the engine, and result in many other problems of the fuel system [1]. The fouling or coking occurs during autoxidation or pyrolysis, respectively. Autoxidation is related to dissolved oxygen, which results in hydroperoxides and other oxidized products, these products are responsible for the formation of solid deposits. For pyrolysis, many researches have been conducted on the pyrolysis mechanism [2–5], the effect factors on decomposition [6–9], the products from decomposition [10–13] and the coke deposition [14–17].

In autoxidative regime, deposit formation behavior is affected by fuel composition, metal surface properties, dissolved oxygen concentration, and the fuel temperature, etc. Zabarnick and Ervin et al. [18–22] carried out lots of studies on the thermal oxidative stability and fouling mechanism of hydrocarbon fuels. They have developed the 21-step pseudo-detailed chemical kinetic model which can predict deposition formation, the consumption of dissolved oxygen, as well as the formation and consumption of hydroperoxides. Larsen et al. [23] found that alkanes and cycloalkanes behaved in a similar manner and were quite reactive, and that polycyclic aromatics were very stable. Zabarnick et al. [24] and Hazlett et al. [25] reported that heteroatoms in fuels, particularly sulfur, influenced the fouling of fuel. The dissolved oxygen concentration is an important parameter for determining the amount of deposits. Zabarnick and Grinstead [26], Heneghan et al. [27], Edwards and Krieger [28] found that the reduction of dissolved oxygen concentration would result in a decrease in deposits. Ervin and Williams [29] found that the deposits in heated sections increased as the oxygen consumption decreased. Roan and Boehman [30] found that removing dissolved oxygen by sparging

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with nitrogen did not useful to reduce the amount of oxidative deposits.

Temperature is an important parameter affecting the rate of fuel oxidation. Hazlett et al. [31] divided the degradation of fuels into autoxidation (<260 °C), pyrolysis (>400 °C) and intermediate regime (260–400 °C) in terms of temperature at which different sets of reactions occur. The formation of oxygenated products is principal in autoxidation, while these oxygenated products primarily decompose in the intermediate regime. The effect of temperature on fouling is complex. Chin and Lefebvre [32] reported that the deposition rate of DF_2 and kerosene increased with fuel temperature and wall temperature. Tevelde and Glickstein [33] studied the thermal stability of JP-5 and three alternative fuels, and the results indicated that the deposition rates of four fuels were sensitive to wall temperature. Balster and Jones [34] reported that the autoxidation rate increased with temperature, but the total deposits did not necessarily increase. Zabarnick et al. [35] suggested that the inverse dependence on temperature was associated with different amounts of dissolved oxygen in fuel. Stiegemeier et al. [36] found that the deposition rates of JP-7 and JP-10 were rather insensitive to wall temperature, while that of RP-1 had the inverse trend. But above all, there are many factors which influence the deposition of fuels. The change of an above effect factor will cause a change in the fouling phenomenon. The fouling phenomenon of different fuel may be greatly different. Therefore, more studies for the influence of the fouling phenomena and wall/fuel temperature on fouling are required.

Now *exo*-tetrahydrodicyclopentadien called JP-10 is an endothermic hydrocarbon fuel, produced synthetically by the hydrogenation of dicyclopentadiene and the catalysis of endo-tetra hydrodicyclopentadien. It is a popular aviation fuel because of high volumetric energy and thermal stability. Especially, the fuel demand of aviation traffic will keep on increasing, while aviation fuel production is predicted to decrease with the decreasing crude oil production [37]. JP-10 as an alternative fuel can play a role in increasing available fuels. Bruno et al. [38] has reported the thermochemical and thermophysical properties of JP-10. Meylemans et al. [39] researched the low-temperature properties of blend of JP-10 and TDF. Coking deposition associated with thermal decomposition of JP-10 has been investigated [6,15]. It was found that metal surface enhanced the thermal decomposition of JP-10 and the carbon deposits of JP-10 were mainly in the form of irregular carbon particles in morphology. Literature about the fouling of JP-10 is scarce and the effect of wall temperature on that is not clear. This paper aims at reporting the experimental data for oxidative deposit of JP-10 and investigating the effect of wall temperature on that, which is helpful to understand the properties of the coking of fuels and develop alternative fuels.

2. Experimental section

2.1. Test apparatus and procedure

The experiments were conducted in a flowing reactor which was shown schematically in Fig. 1. The main components of the flowing reactor were a copper heat exchanger and the test section. The copper heat exchanger which was 500 mm in length and 600 mm in diameter clamped tightly the test section. The test section was a stainless steel tube (China SS316L) which was 3.175 mm in outer diameter, 2.175 mm in inner diameter and 500 mm in length. The test section was heated by eight identically electrical rod heaters which were inserted into the copper heat exchanger. Four electrical rod heaters were evenly arranged along the circumference in one end of the copper heat exchanger, while others were distributed in the same way in the other end. The wall temperature was measured using 9 type-K thermocouples about 5 cm intervals along the tube. The fuel temperatures and pressure at the inlet and the outlet of the test section were also measured using the platinum resistances and pressure transmitters, respectively. In this work, the uniformity of wall temperature can be controlled within 2 °C. Before each experiment, the test tube was cleaned using ultrasonic in alcohol for 20 min, and the flow system was also cleaned by hexane and alcohol, respectively. The system was purged with argon for 20 min to eliminate the effect of oxygen on the experiment results before fuel flows into flow system.

Experiments were conducted for 24 h with a flow rate 0.5 ml/min at the system pressure of 3.84 MPa and wall temperature of 260 °C, 300 °C, 350 °C, respectively. The inlet fuel temperature was 151 ± 2 °C, 173 ± 5 °C, 210 ± 10 °C at three wall temperatures, respectively. The outlet fuel temperature was 247 ± 2 °C, 284 ± 2 °C, 331 ± 2 °C at three wall temperatures, respectively. Before flowing into test section, the fuel was not preheated to simulate the actual operating condition. After the experiment, argon was purged to remove the residual fuel in reaction tube and cool the reaction tube.

2.2. Materials

The sample of JP-10 which purity in mass fraction is 99% was supplied from Liming Research Institute of Chemical Industry, China. The physical properties are listed in Table 1, where ρ is the density, η is the viscosity, T_f is the flash point, T_b is the normal boiling point, and T_c and p_c are the critical temperature and pressure. The chemical compositions of SS316L tube in mass fraction are 0.01% C, 0.67% Mn, 0.032% P, 0.002% S, 0.31% Si, 16.73% Cr, 10.10% Ni, 2.03% Mo, 0.065% N, respectively.

2.3. Analysis of coke deposit

After the experiment, the reaction tube was cut into 10 segments. Each segment was about 50 mm in length. The samples were washed with hexane, and then vacuum dried at 80 °C for two hours. The amount of coke deposition formed on stainless steel surface was measured using a LECO RC612. Temperature-programmed oxidation (TPO) profiles were plotted as the amount of CO_2 evolution versus the furnace temperature. TPO is an important technique to characterize the nature of coke deposition from hydrocarbon fuels [8,16,40,41]. The TPO profiles may give multiple CO_2 evolution peaks, which can provided the information of oxidation reactivity or structure of coke deposition. The samples were heated from 100 °C to 900 °C at a heating rate of 30 °C/min and held at 900 °C for 5 min. The ultra-high purity oxygen at flow rate of 750 ml/min was used in the TPO experiments. The morphologies of surface coke deposition are characterized by a field emission scanning electron microscope (FESEM). The location where the samples are analyzed by FESEM images is at 40–45 cm section. The identical experiments under the same condition were repeatedly conducted to check experimental repeatability. In this work, the Student's *t*-test is used to characterize the results. The reproducibility and relative error of data are evaluated to be 3.5% and 15% at confidence level of 95%.

3. Results and discussion

3.1. Amount of carbon deposit of JP-10

Fig. 2 shows the distribution of coke deposition of JP-10 fuel on the SS316L tube surface along the length of the test section at 3.84 MPa and at wall temperature of 350 °C, 300 °C and 260 °C, with a flow rate 0.5 ml/min for 24 h. It shows that there is a