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Chemical and Physical Properties of Soot as a Function of Fuel Molecular Structure in a Swirl-Stabilized Combustor

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Soot samples were extracted from a swirl-stabilized, liquid-fuel fired laboratory combustor to (i) assess the validity of the ASTM smoke point test in predicting the sooting characteristics of fuels in complex combustion systems and (ii) assess the effect of fuel molecular structure and smoke suppressant additives on the physical and chemical properties of soot. Fuels utilized were shale JP-8, iso-octane, and blends of iso-octane with either decalin, toluene, tetralin, or 1-methylnaphthalene such that smoke points similar to JP-8 were achieved. Each fuel was separately blended with 0.05 percent (by weight) ferrocene smoke suppressant. The results indicate that the ASTM test can be misleading, increased fuel molecular complexity increases soot content of higher molecular weight polycyclic aromatics, and ferrocene additive reduces small particle number density but not necessarily the soot mass loading.

Introduction

With the continued consumption of petroleum resources outpacing discovery, the use of fuels derived from shale and coal is evolving as a practical alternative for jet fuels. Liquid synfuels, however, are typically less volatile and have a lower hydrogen content than petroleum fuels due to their higher aromaticity. The net result is an increased propensity of liquid synfuels to form soot with an attendant increase in flame radiation, deposit of carbonaceous material, and emission of particulate. The widespread use of these synfuels in aircraft propulsion and power generation systems will depend on overcoming the propensity of these fuels to form soot. Approaches under consideration to reduce this soot production include the future development of fuel-flexible gas turbines through combustor modification and the use of smoke suppressant fuel additives for the current generation of gas turbines.

Two questions arise in the resolution of problems associated with the use of liquid synfuels in complex (i.e., recirculating and turbulent) flows typical of gas turbine combustors. First, what are the mechanisms responsible for soot formation and burnout in complex flows? Such information could provide direction for the use of additives in suppressing the soot production in existing engines and identify paths for combustor modification in next generation gas turbines. Second, what methods are appropriate for testing the sooting propensity of fuels in complex flows? In

particular, is the ASTM-D1322 smoke point [1] method a suitable or meaningful test of sooting propensity of jet fuels?

The current interest in the mechanisms of soot formation and burnout is directed to both simple and complex flows, and the results from recent studies are beginning to identify the correspondence between sooting, and fuel properties and flame conditions. For example, in simple diffusion flames, the flame temperature is asserted to be the dominant parameter affecting sooting propensity with fuel molecular structure playing a role only as it affects the flame temperature [2]. In complex flows, the evidence is not yet sufficient to assess the causal/effect relationships. On one hand, soot production has been found to correlate with fuel hydrogen content in a practical combustor [3]. However, an influence of fuel molecular structure has been reported with fuels containing high concentrations of polycyclic aromatics [4]. And the role of aerodynamics in complex flows as it relates to both soot production and perturbation of diagnostics employed for soot measurements is just now being addressed [5].

The soot measurements employed in the conduct of these studies include the gross weight emission and flame radiation. Additional measurements will be both desirable and necessary if the mechanisms governing the formation and burnout of soot in complex flows are to be resolved. In particular, measurements of both the physical properties of soot (e.g., size and morphology) and the chemical properties of soot (e.g., polynuclear aromatic content) are required to assess (i) the chemical mechanisms active in the production of soot, (ii) the radiative properties of the soot, and (iii) the environmental impact associated with the emission of the soot.

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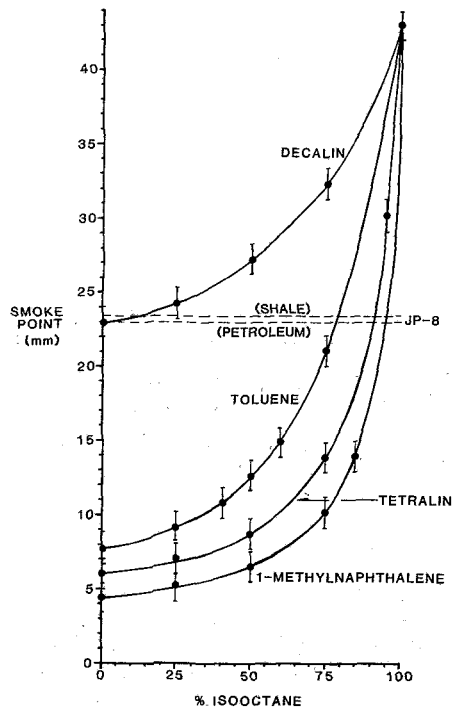


Fig. 1 ASTM smoke point tests

One element of the present study is directed to the acquisition of these data.

The second element of the present study addresses the ASTM-D1322 method historically used to establish the sooting propensity of aviation fuels. Because of the substantial differences in aerodynamics between the ASTM method (which employs a laminar diffusion flame) and a gas turbine (which features a complex flow), the reliability of the method for predicting the relative smoking intensity of different fuels in gas turbine combustors has, for some time, been open to question (e.g., [6]). The advent of syncrudes and their attendant propensity to form soot is renewing interest in this question. Although smoke point has been found to correlate with flame radiation in a practical combustor operated with different fuels [7], gravimetric data must also be obtained for a variety of fuel types, combustor configurations, and combustor operating conditions before the adequacy of the ASTM smoke point method can be fully assessed.

Objectives

The objectives of this study were to assess:

- The effect of fuel molecular structure and the smoke suppressant fuel additive ferrocene on the chemical and physical properties of soot in complex flows
- The validity of the ASTM method in predicting the smoke point of turbine fuels in complex flows

Approach

The present study employed six liquid fuels of varying molecular structure operating in a swirl-stabilized, spray-atomized combustor. Iso-octane served as the baseline fuel as it represents a major component of JP-8 and serves as the reference fuel in the ASTM smoke point test. The five remaining fuels consisted of shale-derived JP-8 and mixtures of iso-octane with either decalin (decahydronaphthalene), toluene, tetralin (1, 2, 3, 4 tetrahydronaphthalene), or 1-

Table 1 Fuel summary

Fuel	Smoke point ^a mm	H wt. %
Iso-octane	43.0	15.79
Shale JP-8	23.0	13.89 ^b
Blend 1	86% decalin 14% iso-octane	24.3
Blend 2	21% toluene 79% iso-octane	24.0
Blend 3	8% tetralin 92% iso-octane	25.3
Blend 4	5% 1-methylnaphthalene 95% iso-octane	22.2

^a Distance above base of burner at which sooting first occurs

^b Wright Patterson AFB

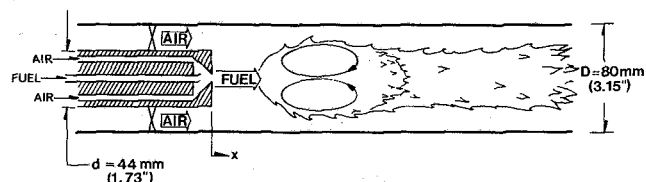


Fig. 2 Combustor configuration

methylnaphthalene. The amount of hydrocarbon blended with the iso-octane was selected to yield the same ASTM smoke point as that attained with the shale derived JP-8. The choice of the pure hydrocarbon liquids represented both a broad variation in fuel molecular structure and classes of compounds found in petroleum and synthetic fuels.

The amount of each compound blended with iso-octane was determined by first preparing a curve of smoke point versus volume percent iso-octane (Fig. 1). Table 1 summarizes the composition and the actual smoke point found for each blend. The smoke points, while not exact, are well within the achievable accuracy of the smoke point test (± 1 mm) shown by the error bands in Fig. 1.

Each fuel was injected as a liquid spray into a complex-flow laboratory combustor. Soot samples were obtained using an extractive probe and subsequently analyzed to determine weight concentration, chemical composition, and morphology. In a second series of tests, a soot suppressant additive (ferrocene) was mixed with each fuel to assess the effect on sooting propensity as a function of fuel molecular structure. A single ferrocene additive concentration of 0.05 percent by weight was selected based on data in Naval Air Propulsion Test Center reports [8] and work conducted by Moses and Naegeli [9].

Facility

The combustor (Fig. 2) was comprised of a quartz tube with an i.d. of 80 mm (3.15 in.). Fuel was injected at the face of a centerbody with a flush-mounted, twin-fluid atomizer (Spraying Systems J-Series, 2050 Fuel Cap, 67147 Air Cap). To create a toroidal recirculation zone, and thereby centralize and remove the region of soot formation from the effect of solid boundaries, swirl vanes with a turning angle of 60 deg (swirl number of 1.4 as calculated in [10]) were located 41.3 mm (1.6 in.) upstream of the centerbody face. The length of the quartz tube was arbitrarily selected at 1.22 m (48.0 in.).

The combustor was operated in the present study at a reference velocity of 15 m/s, an overall equivalence ratio of 0.2, and at atmospheric pressure. The rationale for this rests with the interest in (i) establishing a record of soot production performance at a base condition of atmospheric pressure, and

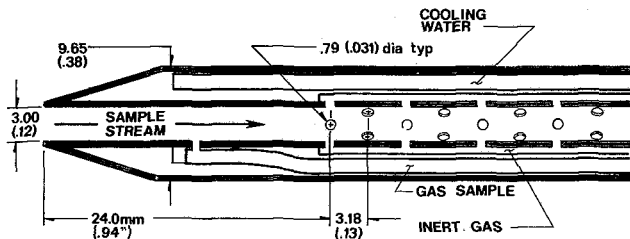


Fig. 3 Extractive probe

(ii) comparing sooting propensity between the ASTM-D1322 smoke point test and a complex flow at a common pressure, the magnitude of which is fixed by the ASTM method.

The sample probe used in this study (Fig. 3) was of stainless steel construction with an overall o.d. of 9.5 mm (3/8 in.) and a 3.0 mm (1/8 in.) i.d. sampling tube. The overall length of the probe was 616 mm (24 1/4 in.), including a large radius bend of 508 mm (20 in.) to clear the combustor exhaust stream. The probe was water cooled to approximately 40°C and was fitted with a gas sampling port for gas analysis and a set of inert gas injection ports. The purpose of the inert gas was to dilute the mixture and thereby quench reactions, lower the dew point of the sample, and prevent deposition of particles along the tube wall. The multiple injection ports served to diffuse the inert gas injection and thereby eliminate any undesirable jetting.

The sample system consisted of a heated sample line leading from the probe to an oven. The oven, heated to 50°C, housed a four-way flow switching valve which permitted sequential filtration between three sample filters and a bypass filter. Two of the filters consisted of 47-mm single stage, Gelman microquartz fiber filters (DOP efficiency of 99.9 percent). These filters were used to collect sample for the weight concentration, chemical, and elemental analysis segments. Downstream of one of the microquartz filters and located outside of the oven was an aluminum chamber containing 20 grams of XAD-2 resin to absorb any vapor phase polycyclic aromatic hydrocarbons (PAH). The third filter used two-stage filtration consisting of 47-mm Nuclepore membrane filters with 5.0-μm and 0.2-μm pore size. Nuclepore filters were selected for their adaptability to scanning electron microscopy.

The sampling location selected for the collection of soot was at an axial location $x/d = 18$ (81.3 cm, 32.0 in.) downstream of the centerbody face and a radial position adjacent to the duct wall 31.8 mm (1.25 in.) from the centerline. The axial location was removed sufficiently from the primary zone to avoid any effect of probe perturbation. The radial location was selected to minimize sampling time by locating the probe at the point of peak soot loading, a condition set up by the swirl-induced transport of soot toward the duct wall.

Analysis

Prior to use, the microquartz filters were dried for 1 hr at 400°C to remove any residual organics and absorbed water. They were subsequently stored in a desiccator until required. A electromicrobalance (Cahn Model 26) was used to weigh the filters before and after the sample collection period, with the filters being desiccated for another 24 hrs prior to reweighing to remove any water absorbed during the collection period. The weight concentration was calculated using the following equation:

$$C = \frac{\text{weight gain}}{\text{net sample volume (STP)}}$$

Approximately 2 mg of sample was collected on the

Table 2 Gravimetric results

Fuel	Smoke point mm	Weight concentration ^a mg/m ³
Iso-octane	43	1.06
86% decalin 14% iso-octane	24.3	5.37'
21% toluene 79% iso-octane	24.0	6.74
8% tetralin 92% iso-octane	25.3	4.10'
5% 1-methyl-naphthalene 95% iso-octane	22.2	5.12
Shale JP-8	23.0	23.52

^a Average of two determinations except where noted with (')

microquartz fiber filter used for PNA analysis. The filter and XAD-2 resin were separately soxhlet-extracted for 20 hrs with 200 ml of methylene chloride. All of the solvents used were distilled in glass purity. The extract was then evaporated to dryness under reduced pressure in a rotary evaporator with the residue resuspended in 10 ml cyclohexane. A dimethylsulfoxide (DMSO) separation scheme [11] was then employed to isolate the PNA component. PNA identification was accomplished with the use of a HP 5830A gas chromatograph and a HP 5992A gas chromatograph/mass spectrometer in conjunction with a J & W 30 M SE-54 capillary column. The column was used in a splitless mode, with an inlet purge of 50:1 and with a helium carrier flow rate of 1 ml/min.

A C-H-N analyser (Perkin Elmer Model 240B) was used to determine the carbon weight percent of the soot particles before and after the PNA extraction step. For those fuels to which ferrocene was added, an absorption spectrophotometer (Perkin Elmer Model 403) was used to determine the amount of iron incorporated into the soot. As this step was performed after the soxhlet extraction, both the organic extraction and insoluble fraction remaining on the filter were analyzed for iron content. Physical characterization of the particles was conducted utilizing a Hitachi 5-500 scanning electron microscope (SEM).

Results

Physical Properties.

Gravimetric Results. The sooting propensity of the fuels under complex flow conditions are presented in Table 2. The soot weight concentrations of iso-octane and the pure fuel blends were consistent with the sooting propensity indicated by the ASTM method, whereas the soot weight concentration of the chemically complex shale JP-8 was significantly higher (>400 percent) than the soot weight concentrations produced by the fuel blends prepared with a similar smoke point.

The ASTM smoke point tests results obtained with the fuels after the addition of ferrocene are presented in Table 3. The additive resulted in an increase in the smoke point in all cases. However, the effectiveness varied with fuel type.

Under complex flow conditions, the weight concentration results obtained with the ferrocene additive (Table 3) divide into two distinct groups – a general *increase* in the particulate weight concentration (pure iso-octane and the fuel blends), and a substantial *reduction* in particulate weight concentration (shale JP-8). In an effort to explain the weight increase on all but the shale JP-8 samples, the amount of iron present in the collected samples was required.

Table 3 Gravimetric results with ferrocene additive

Fuel	With ferrocene additive			Change in	
	Smoke point mm	Uncorrected weight concentration mg/m ³	Corrected weight concentration ^a mg/m ³	Smoke point mm	Weight concentration ^b %
Iso-octane	>50	1.98	1.48	+ >7	0
86% decalin 14% iso-octane	28.3	5.65	5.54	+4.0	0
21% toluene 79% iso-octane	26.3	7.80	7.45	+2.3	0
8% tetralin 92% iso-octane	30.0	6.82	6.00	+5.3	+50%
5% 1-methyl-naphthalene 95% iso-octane	35	8.40	8.12	+12.8	+50%
Shale JP-8	24.7	9.75	9.22	+1.7	-60%

^a Subtracting weight contribution of iron using data of Table 4

^b Within accuracy

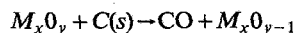
Table 4 Iron content of samples

Fuel	Soluble fraction (soxhlet extracted) μg	Insoluble fraction μg	Iron corrected to iron oxide mg	Total particulate weight collected mg	Corrected particulate weight ^a mg
Iso-octane	<1	166	0.214	1.02	.81
86% decalin 14% iso-octane	<1	191	0.246	2.49	2.34
21% toluene 79% iso-octane	<1	225	0.289	4.47	4.18
8% tetralin 92% iso-octane	<1	223	0.287	3.32	3.03
5% 1-methylnaphthalene 95% iso-octane	<1	402	0.517	5.00	4.48
Shale JP-8	<1	132	0.170	2.66	2.49

^a Total particulate weight collected less iron corrected to iron oxide

The fraction of particulate present after the soxhlet extraction was first examined to assess the extent to which iron was organically complexed. The results are presented in Table 4. In all cases, organically complexed iron accounted for less than 1 μg (< 1 percent by weight of the total measured). To assess the amount of iron contained in the nonorganic fraction, the collected sample that remained after the soxhlet extraction was washed in concentrated nitric acid to remove the nonorganic fraction and the weight of iron was determined from atomic absorption spectrophotometry, corrected to iron oxide, and subsequently subtracted from the gross sample weight to yield the true weight of soot collected. The net result, shown in Table 3, was that no significant difference in soot weight concentration was found between the ferrocene and nonferrocene runs with the pure iso-octane, and the decalin and toluene blends. In direct contrast, the soot weight concentration of the tetralin and 1-methylnaphthalene blends increased 50 percent in the presence of ferrocene, and the soot weight concentration of the chemically complex JP-8 decreased 60 percent.

Ferrocene is thought to first be occluded in the soot particle and act in the later regions of the flame by both lowering the ignition temperature of the soot and acting as an oxidizing agent via the mechanism [12]



The effect of ferrocene is likely a function of temperature, residence time, and chemical structure of the soot matrix.

Recognizing that, in the present study, the temperature field and residence times remain relatively uniform between the different runs (as a result of the consistency in aerodynamics and narrow range of fuel heating values), the gravimetric results suggests that the effect of the ferrocene additive is strongly tied to the chemical structure of the soot matrix which, in turn, is tied to the fuel molecular structure.

Morphology (SEM Analysis). Typical results of the scanning electron microscope (SEM) analysis of the Nuclepore filter samples are shown in Fig. 4. Only the second stage (0.2-μm) filters are shown, as the first stage (5.0-μm) filters experienced no deposition.

All the soot collected was 0.5-1.0 μm agglomerates with either lacy chain or semispherical structure comprised of smaller, individual 0.05-0.1 μm spherules. This is consistent with the structure and size observed in full-scale combustor studies [13, 14], as well as in small-scale lab combustors [6].

The effect of the ferrocene additive becomes apparent upon close examination of Fig. 4. Note the reduction of the number density of the small particulate ($d < 0.1 \mu\text{m}$) in the presence of the additive. A decrease in the number of smaller particles is expected as these would be the first to burnout if, in fact, ferrocene acts to reduce the ignition temperature for oxidation. Such a decrease is also consistent with ferrocene additive studies in full-scale jet engines in which reductions in the number density of small particles have been observed [8].

The SEM results are consistent with and help to interpret

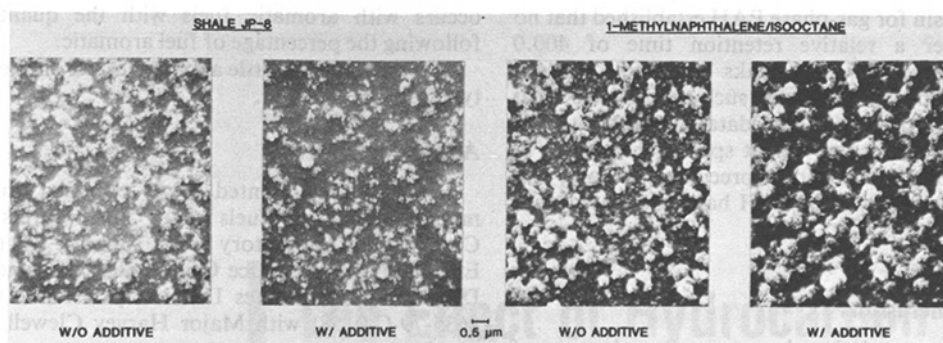


Fig. 4 Particulate morphology

Table 5 Soot PAH content

Compound name	Retention indices ^a	Fuel											
		Isooctane		86% decalin 14% isooctane		21% toluene 79% isooctane		8% tetralin 92% isooctane		5% 1-methyl % isooctane		JP-8	
		w/o add.	w/f add.	w/o add.	w/f add.	w/o add.	w/f add.	w/o add.	w/f add.	w/o add.	w/f add.	w/o add.	w/f add.
Naphthalene	200.00		(3.8) ^g			(0.8)	(0.8)						
	206.67 ± .14 ^b	(4.2)		(0.7)		(0.3)				(0.4)			
	252.73 ± .25 ^c					(0.2)							
	285.98 ± .29 ^c		(50.2)		(8.8)		(1.1)						
	290.98 ± .01 ^c		(3.7)				(11.6)		(0.7)				
Phenanthrene	298.75 ± .24 ^c				(4.4)		(0.6)						(3.3)
	300.00		(63.0)			(7.1)				(1.0)			
	303.32 ± 0 ^c												(0.5)
	318.15 ± .09 ^c				(6.4)		(10.6)						
	343.79 ± .0 ^c						(0.8)						
Chrysene	348.77 ± .11 ^c		(6.0)		(1.0)		(0.6)						
	396.90 ± .20 ^d					(1.4)	(1.4)	(1.4)	(1.3)	(1.7)	(1.4)	(0.9)	(1.8)
	400.00		(39.0)		(3.9)	(4.4)	(2.2)	(1.0)	(1.0)	(0.6)	(1.4)	(0.8)	(4.0)
	407.40 ± .04 ^d						(0.6)					(0.2)	
	445.99 ± 0 ^d												
Benzo[ghi]Perylene	451.47 ± .19 ^e							(0.7)					
	452.99 ± .21 ^e	(4.0)				(3.8)	(0.9)	(5.1)	(1.4)	(2.0)	(1.7)	(0.8)	
	483.56 ± 0 ^e				(1.6)								
	491.69 ± .13 ^e					(1.4)		(1.6)		(0.6)			
	500.00	(5.5)	(20.3)		(2.6)	(4.7)	(2.0)	(4.3)	(2.8)	(2.2)	(1.5)	(0.4)	(2.7)
	503.18 ± .15 ^e					(0.7)		(1.9)		(0.6)			

^a Retention indices are the relative positions of the compounds retention times to the PAH Standards. The PAH standards are assigned values corresponding to the number of aromatic rings in the standard.

^b Reported as naphthalene equivalent

^c Reported as phenanthrene equivalent

^d Reported as chrysene equivalent

^e Reported as benzo[ghi]perylene equivalent

^f DMSO separation procedure not carried out on these runs (no interfering aliphatic peaks).

^g Number in parenthesis = μ g PNA/mg Soot/M³ recovered; blank indicates no evidence of species

the gravimetric data of Table 3. The weight concentration will primarily depend on agglomerate size and number density with little effect exerted by the number density of the small particulate. As a result, the effect of ferrocene on weight concentration (Table 3) should be reflected by the effect of ferrocene on the agglomerate. This is, in fact, the case. The ferrocene had little impact on the size and number density for iso-octane and the decalin and toluene blends. The soot agglomerates of the JP-8, however, were substantially reduced in size (Fig. 4), which corresponds with the reduced weight concentration found with the presence of the ferrocene. The agglomerate size as well as the number density were substantially increased in the case of the double-ring fuel blends, tetralin, and 1-methynaphthalene, which again parallels the gravimetric data.

Chemical Properties. Gas chromatographic analysis of the methylene chloride extracts identified the presence of a variety of polycyclic aromatic hydrocarbons (PAH) in the extracted soot samples. In order that the peaks from different

chromatograms could be interrelated with this and future studies, relative retention indices scheme developed for PAH was used [15]. A tabulation of the compounds found with the various fuels is presented in Table 5. The presence of aromatic groups in the fuel is shown to carry over to the chemical structure of the soot matrix. For example, the toluene blend (21 percent by volume) contained the largest amount of aromatics which is reflected in the amount of PAH found with the soot. A trend toward higher molecular weight PAH is also evident with an increase in the complexity of the fuel molecular structure (reading table from top left to bottom right).

Integration of the chromatographic peaks and calibration with known amounts of four PAH allowed a quantitative determination of the PAH present. It is noteworthy that small quantities of high molecular weight PAH are present with even iso-octane and decalin, although there are far fewer species present at a significant level ($> 1 \mu\text{g}$). The presence of the ferrocene additive had little effect on the quantities and types of PAH present.

Analysis of the resin for gas-phase PAH established that no peaks evolved after a relative retention time of 400.0 (chrysene). The majority of peaks evolved between naphthalene and phenanthrene in such abundance that resolution was compromised. These data suggest that (i) a large number of low molecular weight species are present in the vapor phase, possibly as residual precursors to soot, and (ii) all the high molecular weight PAH have condensed onto the soot particulate.

Summary and Conclusions

The present study was designed to assess the adequacy of the ASTM smoke point method in characterizing the sooting propensity of fuels in complex flows, and to initiate an impact assessment of fuel molecular structure on the physical and chemical properties of soot. The conclusions derived from the results are as follows:

1 The ASTM smoke point method can be misleading in the prediction of the sooting tendency associated with fuels burned in complex flows. Mixtures of pure aromatic hydrocarbons in iso-octane, blended to produce the same ASTM smoke point as shale JP-8, produced similar soot loadings in a complex flow. The chemically complex JP-8, however, produced soot concentration a factor of four higher in the same flow. The inconsistency between the ASTM test and the complex flow results is attributed to the dependency of sooting on flame temperature and residence time on the one hand, and the role of aerodynamics dictating the turbulent diffusion of oxygen, the temperature field, and distribution of residence times in a complex flow field.

2 The ASTM smoke point failed also to predict the effect of a ferrocene additive in the complex flow. The smoke point increased for all fuels and blends tested indicating a reduction in soot propensity. In the complex flow, the ferrocene additive was found to be effective in reducing only the soot concentration of shale JP-8. In the remaining cases, ferrocene had either no effect or a significant adverse effect. The chemical structure of the soot matrix appears to govern the effectiveness of the iron additive in oxidizing the soot.

3 The soot morphology exhibited the classical size (0.5–1.0 μm) and structure (agglomerates of 0.05–0.1 μm). In the presence of ferrocene, the number density of the smaller particles was reduced. The effect of ferrocene on the agglomerates, however, depended on fuel type. For the chemically complex JP-8, the size of agglomerates was reduced. For two double-ring aromatic blends, both the agglomerate size and number density were increased with an associated impact on weight concentration.

4 A greater proliferation of PAH species in the soot

occurs with aromatic fuels with the quantities of PAH following the percentage of fuel aromatic.

5 Ferrocene has little apparent effect on the quantities and types of PAH present.

Acknowledgment

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