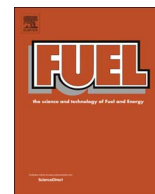




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Effects of aromatic fluids on properties and stability of alternative marine diesels

Jinxia Fu*, Scott Q. Turn

Hawaii Natural Energy Institute, University of Hawaii, Honolulu, HI 96822, USA

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ABSTRACT

Hydroprocessed renewable diesel (HRD-76) and synthesized isoparaffin (SIP-76) have been produced in sufficient quantity for testing by the US Navy, and demonstrated as compatible replacements or blend stock with marine diesel (NATO F-76). Operational limitations with respect to the lubricity, seal swell and cold-flow properties, however, need to be addressed before using 100% HRD-76 or SIP-76. Aromatics are known to improve seal swell and low-temperature characteristics. In this study, three commercial aromatic fluids (Aromatic 100, 150 and 200) produced from petroleum refining were added to HRD-76 and SIP-76 at various concentrations to investigate their impacts on the properties of the biofuel blend. The composition, hydrogen content and physicochemical properties of these aromatic fluids were determined according to ASTM methods and compared with that of biofuels and F-76. The seal-swell capability of the aromatic fluids and biofuel blends was investigated using nitrile O-rings, while the effects of aromatic fluids on fuel low-temperature quality were evaluated using differential scanning calorimetry. In addition, ASTM D5304 and D2274 tests were conducted to investigate the impacts of adding aromatics to biofuels on long-term storage and oxidative stabilities. Although the composition and characteristics of these three aromatics fluids varied, all were capable of inducing o-ring seal swell and adjusting the density of biofuels without significantly affecting the biofuels' stabilities. HRD-76 and aromatic fluid blends also exhibited liquid to solid phase transitions at lower temperatures and greater hydrophilicity than neat HRD-76.

1. Introduction

The US military is the largest single energy consumer in America [1]. Interest by the United States Department of Defense (DOD) in development and approval of alternative fuels produced from renewable sources has grown significantly during recent years driven by the concerns of energy security, cost volatility, and environmental sustainability [2]. Alternative fuels are being sought as drop-in replacements requiring no modification to existing equipment and fuel handling and transportation systems and must possess similar or better qualities compared with equivalent petroleum fuels such as NATO F-76. Extensive laboratory tests have resulted in the approval of hydroprocessed renewable diesel (HRD-76) and synthesized isoparaffin (SIP-76), also known as direct sugar to hydrocarbon (DSH-76). The US Navy has identified a blend of 50% F-76 and 50% HRD-76 or SIP-76 to meet the alternative fuel requirements [3,4]. SIP-76 was also selected as a blending feedstock (up to 10% by volume) in military aviation fuel JP-5 [5]. HRD-76 and SIP-76 are predominantly n- and branched alkanes and contain minima aromatic and heteroatomic compounds [6]. Thus,

neat SIP-76 and HRD-76 exhibit favorable characteristics, such as increased fuel stability and reductions in particulate matter and gaseous combustion emissions, which are attributed to the lack of aromatics [7–9]. The aromatics, however, are believed to provide sufficient material compatibility/seal swell and other “fit-for-purpose” (FFP) properties. [2,9–14]

Aromatics are chemical compounds that contain one or more rings that are planar and have a cyclic arrangement of p orbitals with $4n + 2\pi$ electrons, and each atom of the aromatic ring has a p orbital [15]. In general, fossil fuel is the main natural source of aromatic systems. Traditionally, the source of aromatics were coal tar, a by-product of coke production in the steel industry, and aromatics was obtained by distillation [16]. Now, the major source of aromatics is petroleum and petroleum fuels usually contain paraffinic chains, naphthene rings, and aromatic rings. Aromatic fluid products are marketed as heavy aromatic grades with high solvency and controlled evaporation characteristics [17]. These aromatic fluids are produced from distillation of aromatic streams derived from crude oil and the aromatic content is typically $> 99\%$. The applications of aromatic fluids include agriculture

* Corresponding author.

E-mail addresses: JinxiaFu@hawaii.edu (J. Fu), sturn@hawaii.edu (S.Q. Turn).

chemicals, coatings, industrial cleaners, sealants, process fluids, and refining, fuel, and lube additives. Global production exceeded 454 million kilogram in 2005 [17].

Although HRD-76 and SIP-76 has already been employed as blends with F-76 to power Navy surface ships [4], these two neat biofuels cannot be directly utilized to completely replace petroleum fuel due to the lack of aromatics. The overall impacts of aromatics on fuel storage and oxidation stability have not yet been investigated. An improved understanding of the effect of the type and concentration of aromatics on fuel physicochemical properties and stabilities will assist in identifying potential aromatic candidates to meet fuel system operating requirements. The purpose of this study was to investigate the impacts of aromatics on biofuel properties. Three commercial aromatic fluids, i.e. Aromatic 100 (A100), Aromatic 150 (A150) and Aromatic 200 (A200), were added into HRD-76 and SIP-76 to assess their effects on fuel characteristics, such as physicochemical properties, low-temperature qualities, material compatibilities, water content, and storage and oxidation stabilities. ASTM D5304 [18] and ASTM D2274 [19] methods were employed to investigate the storage and oxidation stability of biofuel blends with aromatics. The fuel blend properties, such as density, viscosity, peroxide value, heat of combustion and acid number, were measured according to ASTM methods.

2. Materials and methods

2.1. Materials

SIP-76. and HRD-76 were provided by Naval Fuels and Lubricants Cross Function Team at Patuxent River, Maryland, United States (PAX River). The SIP-76 and HRD-76 fuel lots were synthesized by Amyris Biotechnologies and UOP, respectively. Aromatic 100, 150, and 200 were produced by ExxonMobil and purchased from Fisher Scientific. The vapor pressure of these three fluids is ranked: A100 > A150 > A200. The primary composition of A100 is C9–C10 alkylbenzenes, whereas A150 and A200 are mainly composed of C10–C12 alkyl benzenes and alkyl naphthalenes, respectively. Thus, A100 is usually recognized as light aromatic, while A150 and A200 are considered as heavy aromatics [17]. The fuels and aromatic fluids were used as received, unless otherwise noted.

2.2. Physicochemical properties

The composition of aromatic fluids, SIP-76 and CHCD-76 was determined by gas chromatography/mass spectrometry (GC/MS). 1 μ L fuel samples were dissolved in 1.5 ml hexane and analyzed using a Bruker 436-GC gas chromatograph and SCION-MS select, single quadrupole mass spectrometer (Bruker Corp., Billerica, MA). The GC was equipped with an Agilent DB1701 column (low/mid polarity, 60 m, (14%-cyanopropyl-phenyl)-methylpolysiloxane), with a 15 m guard-column before the back flush valve, and operated at a helium flow rate of 1.5 ml/min. Hydrocarbon and aromatic reference standards purchased from AccuStandard (AccuStandard, New Haven, CT) and Sigma-Aldrich were used for chemical identification and composition determination. A LECO CHN628 (LECO Corp., St. Joseph, MI) was employed for the carbon and hydrogen content determination of aromatics, biofuels, and their blends. The furnace and afterburner temperature of the system was set at 950 and 850 $^{\circ}$ C, respectively. Approximately 50 mg of each material was placed in a tin foil cup and then covered with \sim 200 mg Com-Aid (> 99% Al₂O₃, LECO Corp., Part No. 501-427) and sealed. The H/C was calculated based on the carbon and hydrogen analysis results.

An Anton Paar SVM3000 Stabinger Viscometer (Anton Paar USA Inc., Ashland, VA) was used to measure the viscosity and density of aromatic fluids and fuel samples at temperatures according to ASTM D7042. The accuracy of the viscometer was tested with certified viscosity standard oil (APN26, Paragon Scientific Limited, UK) and the

measurement repeatability was \pm 0.1% of reading for viscosity, \pm 0.0002 g/cm³ for density, and \pm 0.005 $^{\circ}$ C for temperature. A Parr 6200 Isoperibol Calorimeter (Parr Instrument Company, Moline, IL) was used to measure heats of combustion, i.e. heating values, based on the ASTM D4809 method [20]. The heat of combustion reported in this study is the high heating value (HHV). ASTM D3703 [21] and ASTM D974 [22] were employed to determine the peroxide value (PV) and acid number (AN) of fuels, respectively.

Differential scanning calorimetry (DSC) analyses were conducted using a TA Q2000 system (TA Instruments, New Castle, DE) equipped with an RCS90 temperature control which permits operation over the temperature range of -90 to 400 $^{\circ}$ C. A Tzero low-mass, hermetically sealed pan (TA Instruments, Part No. 901684.901 (lids) and 901670.901 (pans)) was selected for analyses with a sample mass of 2–3 mg. A purge gas of ultra-high purity nitrogen with a flow rate of 50.00 ml/min was regulated by a mass flow controller. Cooling and heating scans for fuel samples were conducted with the following program: (1) equilibrate at set temperature (0 or 20 $^{\circ}$ C), cool at 5 $^{\circ}$ C/min to -90 $^{\circ}$ C, and hold isothermally for 3 min; (2) heat at 5 $^{\circ}$ C/min to set temperature, and hold isothermally for 3 min; (3) repeat step (1) and (2). The step (3), repeated cooling and heating scan, is conducted to ensure that the pan is well sealed and the curve is repeatable. The cooling scan was analyzed to determine the crystallization onset temperature (FO) and the crystallization peak temperature (FP), which reflect the low-temperature quality of fuels.

The water content of the aromatic fluids and biofuels was determined using a DL31 Volumetric Karl Fischer Titrator (Mettler Toledo, Columbus, OH). The titration was conducted by using AQUASTAR Titrant 2 (MilliporeSigma, St. Louis, MO) as titrant, and AQUASTAR Solvent oils & fat (MilliporeSigma, St. Louis, MO) and chloroform (4:1 v/v) as titration medium. Approximately 50 ml of titration medium was first pumped into the cell and titrated dry by means of the titrant. Then \sim 5 g sample was added into the cell with a 6 ml syringe and the titration is started. The sample weight was determined by weighing of syringe before and after injection. The water saturated fuel samples were prepared by mixing equal volume of deionized water and fuel in an Erlenmeyer flask. The flask was then capped with a glass stopper and placed in a New Brunswick I24 incubated shaker (New Brunswick Scientific, Edison, NJ) set at 25 $^{\circ}$ C and 200 rpm for 24 h. The fuel sample was then separated from the deionized water using a separatory funnel and analyzed for water content.

2.3. Material compatibility

The solvent-like interactions of fuel samples with polymeric materials were determined by investigating the overall volume swell characteristics of the fuels. The volume swell testing was conducted using Nitrile rubber, the most widely used elastomer in the seal and O-ring industry with excellent resistance to petroleum based products. An AS001-70 N Nitrile O-ring (Radial Cross Section (CS) 1 mm \times Inside Diameter (ID) 0.74 mm, purchased from TheRubberStore.com) was immersed in a reservoir with 6 ml of test fuel. After immersion in the fuel, the O-ring was immediately photographed every 5 min for the next 2 h using an Olympus digital microscope. At 2 h total elapsed time, the samples were photographed every 30 min for the next 22 h. The diameter of the O-ring was determined using a commercial image processing system Infinity Analyze (Lumenera Corporation, Canada). Assuming isotropic swelling, the change in diameter is proportional to the change in volume as

$$\mu_i = \left[\left(\frac{d_i}{d_0} \right)^3 - 1 \right] \times 100\% \quad (1)$$

where μ_i is the volume swell (%) and d_i and d_0 are the outer diameters of the O-rings in image i and 0 (the reference image taken at time zero). The diameter of each O-ring was measured by the software three times

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