FINAL REPORT

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Diesel Glycerin Emulsion Fuel Project

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Diesel Engine Emissions/Performance of Emulsion Marine Fuels

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Abstract

The work of the Marine Engine Testing and Emissions Laboratory (METEL) at Maine Maritime Academy (MMA) in the area of emulsion biofuels is presented. An overview of METEL is discussed including its unique capabilities in the fabrication, characterization and engine testing of emulsion fuels. The engine emissions test facilities and protocols are discussed including state of the art emissions measurement techniques used to analyze stationary high-speed diesel engines, on board vessel emissions and in METELs medium speed heavy fuel testing facility.

In this paper we describe the emissions of glycerol/diesel and glycerol/biodiesel emulsion blends on marine diesel engines. In addition, fuel physicochemical characteristics and handling issues for these fuels are discussed. Glycerol is an attractive fuel blending material as it is a waste product of biodiesel production with desirable combustion and fuel properties when emulsified into diesel and biodiesel fuels. The cost and carbon reduction benefits of glycerol also make it economically attractive as a fuel component. It is shown that glycerol emulsion fuels exhibit long term shelf stability and when burned can reduce the unwanted pollutants NO_X and THC. Particulate matter emissions are nearly equivalent by mass, but particulate number counts are significantly increased trending towards smaller particles sizes. CO emissions are shown to increase slightly with increasing emulsion concentrations. Data on the performance of emulsion fuels from both laboratory stationary diesels and on board the research vessel *Quickwater* are presented and the operational aspect of using these fuels on vessels is discussed.

Keywords: Glycerol, Glycerin, Glycerol/Diesel fuel, Emulsion fuel, renewable fuel, emissions, marine diesel engines, alternative fuels

1. Introduction

The health and environmental effects of emissions from combustion sources is an important focus of the scientific community [1-3]. Emissions from the marine industry account for approximately 3% of total global green house gas emissions while contributing 15% of global NOx, 13% of global SO_x, and 20% of global particulate matter emissions [4,5]. The International Maritime Organization (IMO) in 2005 through the International Convention for the Prevention of Pollution from Ships (MARPOL) implemented international regulations capping NO_x emissions from international vessels by category and build date along with particulate matter (PM) in Emission Control Areas (ECAs). Additional efforts by the United States included the Act to Prevent Pollution from Ships (APPS) which applied the MARPOL regulations to all applicable vessels operating in the navigable waters of the United States. EPA regulations further capped emissions of oxides of nitrogen (NO_x), total hydrocarbons (THC), carbon monoxide (CO), and total particulate matter (PM) from U.S. flagged vessels by engine category and build date. All above policies stipulate a 3-tiered structure of increasingly stringent emissions limits with the third tier generally taking effect between 2016 and 2018 in U.S. waters and other designated Emissions Control Areas (ECAs). As such, these impending emissions limits and any future policies identify clear openings in the market for drop in fuels, pretreatment, and after treatment technologies to reduce emissions from new engines or subsequent to major vessel conversions and overhauls. Drop-in fuels are appealing as they can be sourced from cost effective feedstock and require little-to-no capital investment by the end user. Glycerol is an attractive feedstock as it is an inexpensive waste product of biodiesel production with desirable combustion and fuel properties when emulsified into diesel and biodiesel fuels [6]. Eaton *et al.* showed that incorporating glycerol into diesel fuel as an emulsion retains many of the diesel fuels original flow properties, such as viscosity and surface tension, but can reduce THC, NO_x and PM emissions significantly. This allows for glycerol, a water-soluble energy source, to be compatible with diesel power applications without the need for engine modification. The use of glycerol has the potential to offset petroleum consumption, reduce diesel emissions and increase the economics of biodiesel fuel manufacturing. Despite these benefits, little is currently understood about the application of glycerol emulsion fuels in the marine environment and their durability across multiple engine platforms. This paper presents the production and test results of glycerol emulsion fuels in the laboratory and on board the research vessel *Quickwater*. Glycerol emulsion fuels containing both ULSD and biodiesel are considered.

2. METEL Overview: Facilities and Capabilities

The Marine Engine Testing and Emissions Laboratory (METEL), established in 2013, is a Tier I University Research Center under the U.S. Department of Transportation Research and Innovative Technology Administration as a collaboration between Maine Maritime Academy and the University of Maine. The focus of the laboratory is on environmental sustainability and is dedicated to research, development, and commercial implementation of viable technologies that improve heavy diesel engine emissions and efficiency. METEL conducts internal research and development projects, while also offering third party testing and evaluation services to industry. Engine test stands of various size engines are housed at METEL's facilities, including a 5.2 kW Hatz 1B30 single cylinder diesel engine, a 27 kW Caterpillar C2.2 marine diesel generator, a 186 kW Cummins QSB6.7, and a 246 kW John Deere 6081AFM75. METEL also operates an instrumented research vessel, the R/V *Quickwater*, a 41ft U.S. Coast Guard cutter outfitted with two 268 kW Cummins VT903 marine diesel engines. The unique research asset allows for testing of fuels, load cycles, and emissions in a real world environment. Construction of a new medium speed engine laboratory is currently in progress. The 1,020kW Wartsila 6L20 generator test cell will be capable of

operating on heavy fuels and will simulate a marine support system with tankage, purifiers, and fuel changeover systems.

METEL has additionally developed capabilities and procedures to characterize fuels and blend various alternative fuels. METEL houses state-of-the-art continuous emissions monitoring equipment for both gaseous and particulate emissions including in cylinder monitoring equipment for analysis of fuel heat release. In order to characterize test fuels, METEL uses ASTM standards to determine the fuel's physical characteristics such as the heat content of the fuel, flash point, viscosity, conductivity, and filter plugging. In the case of emulsion fuels, METEL also employs optical particle sizing equipment to determine the size and distribution of fuel additive or emulsion particles.

3. Experimental Description

3.1 Fuel Composition and Preparation

Fuel components consisted of 2007 certification low-sulfur diesel (ULS) obtained from Chevron Phillips Chemicals (Lot 14GPUL701), waste vegetable oil-derived biodiesel blended between 0-50% (vol/vol), glycerol (99.7 wt% purity from KIC Chemicals, NJ), tap water, Span 80 and Tween 80 surfactants (99 wt% purity from Croda Chemicals). Emulsions were prepared batch-wise in a 50 gallon drum by combing components by weight as listed in Table 1. Glycerol and water were blended and added to the solution of diesel and surfactants. The batch was blended to create a macro-emulsion using a barrel mixer. A micro-emulsion was produced using a Model A dual-feed edge-tone resonant homogenizer (sonolator) manufactured by Sonic corp. The sonolator was operated in continuous mode at a feed rate of 6.7 kg/min using an orifice with a 1.8 mm (0.07 in.) slot width equating to a 206 bar (3,000 psig) line pressure. Emulsion fuel was produced in a single continuous run.

Table 1: Glycerol/ULSD emulsion fuel composition

Component	Weight (%)
2007 Certification ULS Diesel	61.9
Glycerol	28
Water	7
Tween 80	1.4
Span 80	1.2
Ethyl hexyl nitrate (cetane improver)	0.5

After emulsification, ethyl hexyl nitrate was added to achieve 5,000 ppm (wt/wt). The resulting fuel properties are shown in Table 2. The emulsion is considerably denser than the base 2007 ULS diesel due to the heavy components (glycerol/water), which follows a nearly linear proportion increase with increasing emulsion concentration. Conversely, kinematic viscosity is shown to follow a non-linear relationship with increasing emulsion concentration. For the emulsion fuel produced, the kinematic viscosity was determined to be 13.9 cSt indicating that fuel handling and injection operations should not require modifications for operations. The emulsion fuel also requires a lower stoichiometric air/fuel ratio indicating that diesel operation within stock engine configuration will be lean which should improve combustion efficiency.

Table 2: Relevant physicochemical properties of emulsion fuel and its primary components.

Property	ASTM Method	2007 ULS Diesel	Glycerol	Emulsion Fuel
Specific Gravity @ 25°C	D4052	0.8423	1.251	0.9178
Derived Cetane Number (IQT)	D6890	47	--	31.5
Viscosity @ 40°C (cSt.)	D445	2.2	225.8	13.9
Net heat of combustion (MJ/kg)	D3338	42.9	16	32.1
Hydrogen (wt%)	D3343	13.2	8.8	11.7
Carbon (wt%)	(Calculated)	86.8	39.1	66.7
Oxygen (wt%)	(Calculated)	0	52.1	21.6
Air/Fuel Ratio (Stoich)	(Calculated)	14.41	7.45	11.63

A Malvern Zetasizer was used to determine the dispersion and droplet distribution of the resulting fuel. Stability is an important property for transportation fuels for storage and distribution. To determine the suitability for the emulsion fuel for transportation applications, the dispersion and droplet size was tracked over a period of 1 month to quantify aging kinetics. In these tests, 250 mL of sample was obtained off the production stream and stored in a glass jar. On a semi-weekly basis, the sample was re-agitated by hand and approximately 3 mL of sample was removed for dispersion analysis. Figure 1 compares the number distribution of emulsion droplets as produced and after 8 days and 30 days of aging. The freshly produced emulsion exhibits a multi-modal distribution with the first mode occurring between 200 and 700 nm and second occurring between 800 nm and 3,100 nm. The first mode is consistent with surfactant micelles while the second is consistent with micro-emulsion formation. The freshly produced fuel had a mean droplet diameter of 1,142 nm. The samples analyzed after 8 and 30 days of aging show the presence of ripening reaching a stable, single-mode distribution with 2,204 and 2,437 nm mean droplet diameters, respectively. This result indicates that equilibrium is obtained with droplets in the micrometer range.

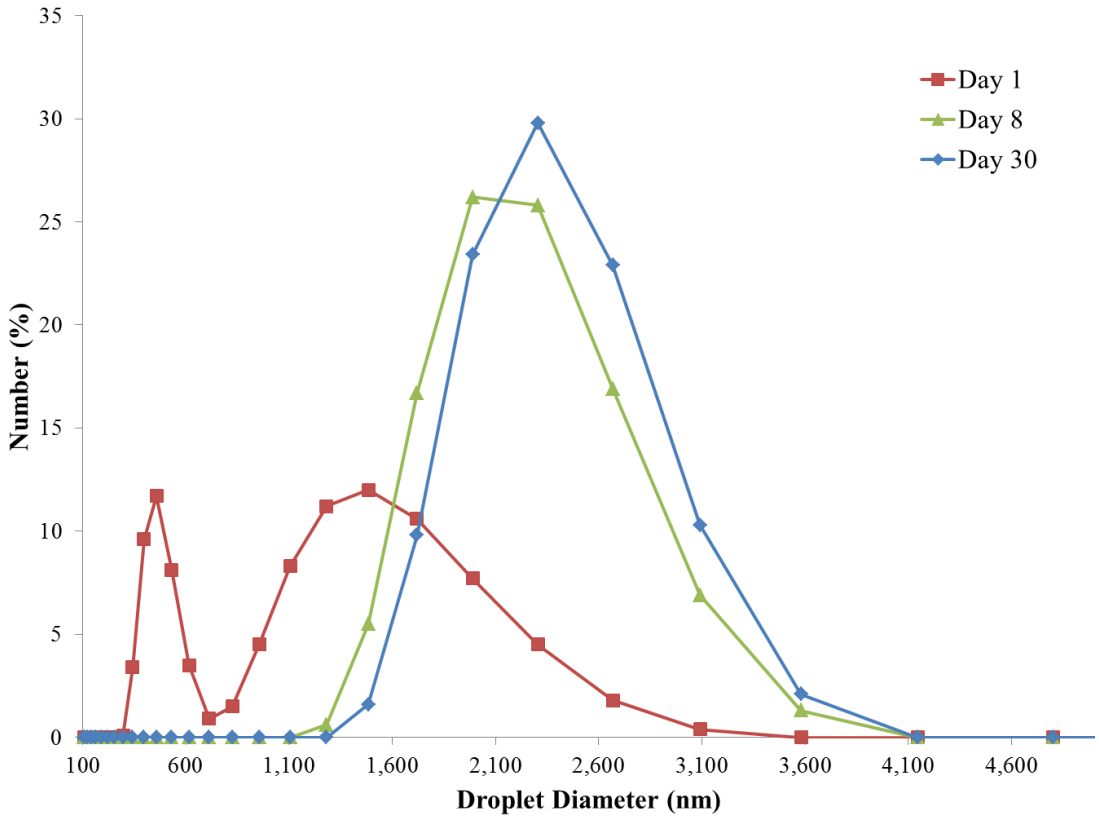


Figure 1: Number distribution of emulsion droplets measured with a Malvern Zetasizer as produced and after 8 days and 30 days of aging.

Figure 2 presents the growth of mean droplet diameter as a function of time. Average particles diameters are shown to increase over the first week of aging reaching a meta-stable droplet size exceeding 2,000 nm. The observations are well captured by a logarithmic rate. To quantify the rate of droplet ripening, the data was fit to a simple step-response model as shown in Equation 1.

$$S(t) = S_{\infty}\left(1 - e^{\frac{-t}{\tau}}\right) \qquad \text{Eq. 1}$$

where S(t) is the mean droplet diameter, S_{∞} is equilibrium droplet diameter (taken at 30 days) and τ is the time constant. The rate constant was determined by linear regression and found to be 12.4 days, which provided an adjusted coefficient of determination of 0.56. The fuel stability analysis provides strong support for the use of emulsion fuels in transportation applications.

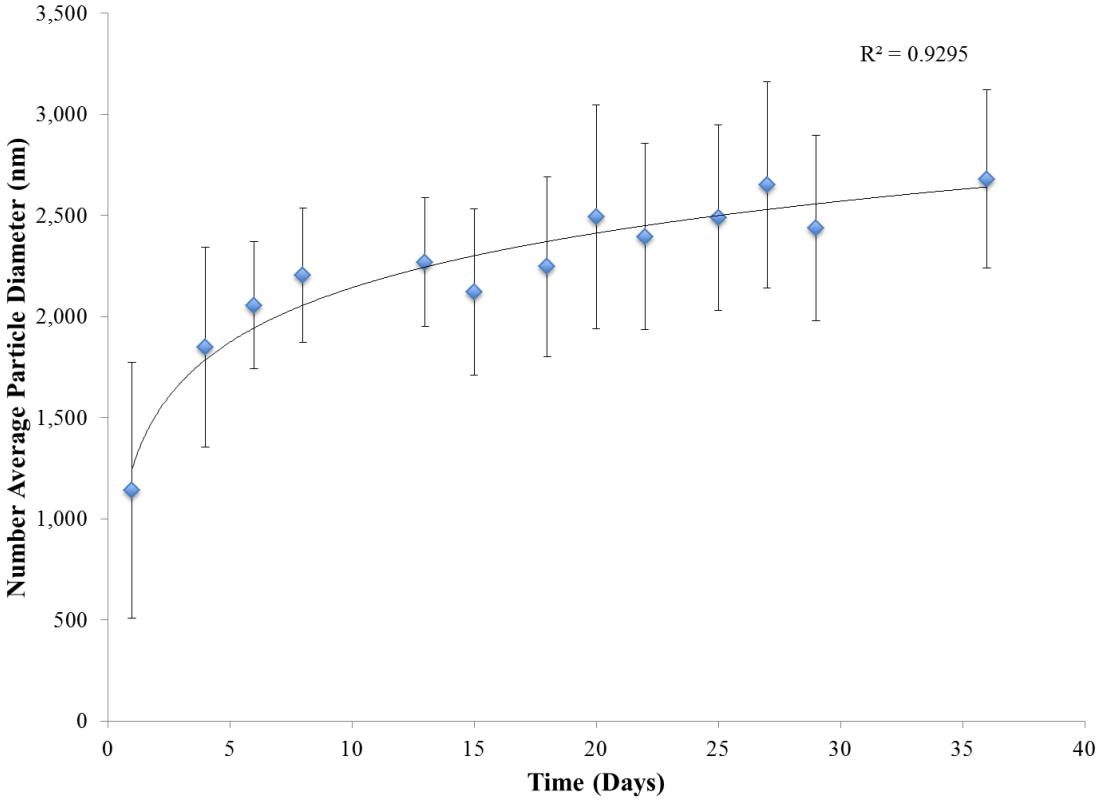


Figure 2: Growth of mean droplet diameter as a function of time.

3.2 Combustion Test Equipment

Stationary engine fuel testing and emissions measurements were conducted on a laboratory housed CAT C2.2 marine diesel generator test cell. The engine is a 4-cylinder indirect injection turbocharged diesel with a bore of 84 mm and stroke of 100 mm. The engine operates at 1,800 RPM with a maximum power rating of 27 kW. The engine test cell was instrumented with a variable frequency drive (VFD), resistive load bank, current, and voltage sensors to set and measure load on the engine. Fuel flow is measured gravimetrically via Omega LCR-50 load cells. Intake air mass flow rate and inlet air temperature are measured with a mass airflow sensor from PMAS with 0.25% measurement uncertainty and 0.4% repeatability. Exhaust emissions ports were located several pipe diameters downstream of the turbine housing of the turbocharger. The duty cycle utilized on the CAT C2.2 test cell conformed to ISO 8178 standards and consisted of starting at idle,

100% rated engine load, 75%, 50%, 25%, 10%, and back to an idle. All load settings were maintained for a sufficient duration to achieve steady state.

Marine environment fuel testing and emissions measurements were conducted on the research vessel *Quickwater*. The *Quickwater* is a 41ft. Coast Guard cutter-class workboat equipped with two VT903 360 hp (268 kW) Cummins marine diesel engines. The dual propeller shafts were instrumented with strain gauges and Datum Electronics shaft power measurement kits to measure shaft torque, RPM, and power. Fuel flow is measured via Kral OMX-20 flow meters. Intake air mass flow rate and inlet air temperature are measured with a mass airflow sensor from PMAS with 0.25% measurement uncertainty and 0.4% repeatability. The exhaust from the port engine of the vessel was outfitted with sampling lines for monitoring gaseous and soot emissions. The sampling ports were placed 0.6 meters after the turbine housing of the turbocharger and before the water-jacketed portion of the exhaust. The duty cycle utilized on *Quickwater* conformed to ISO 8178 standards and consisted of starting idle in gear, 100% rated engine load, 75%, 50%, 25%, and back to an idle in gear. All load settings were maintained for a sufficient duration to achieve steady state.

Dedicated emissions monitoring equipment used during all testing included a MKS 2030 FTIR with heated sampling equipment for gaseous emissions measurements, and a BMI 1710 Mixing Condensation Particle Counter (MCPC) for soot number concentration emissions measurements. The MCPC was additionally equipped with a heated dual stage ejector pump and calibrated critical flow orifice dilution system operating at 150 Celsius and a dilution ratio of approximately 1000. All emissions and engine specific performance monitoring equipment was controlled and recorded with LabVIEW.

4. Results

4.1 Glycerol/Diesel Emulsion

The glycerol/diesel emulsion fuel consisting of components outlined in Table 1 was tested for fuel performance and emissions in comparison to diesel fuel. Figure 1 illustrates gaseous and particulate emissions collected on the CAT C2.2 marine diesel generator test cell for diesel and the glycerol/diesel emulsion fuel. Figure 3a illustrates energy weighted emissions of NO_x + THC with a reduction in emissions evident for the emulsion fuel over all load settings. Figure 3b shows energy weighted emissions of CO significantly elevated for the emulsion fuel over all load settings. The significant variation in CO emissions may be due to incomplete combustion or changes in chemical kinetic pathways due to changes in molecular composition of the fuel [7]. Figure 3c illustrates energy weighted emissions of soot particulate count. The data depicts consistently elevated number concentrations of soot particulates for the emulsion fuel. Similar results were reported in literature for other oxygenated fuels [8]. Fuel consumption as a function of generator output power is illustrated in Figure 3d. The emulsion fuel exhibits a lower energy density and as a consequence results in an increase in fuel consumption. Thermodynamic efficiency exhibits little

dependence on fuel type despite the change in fuel chemistry as shown in Figure 3e.

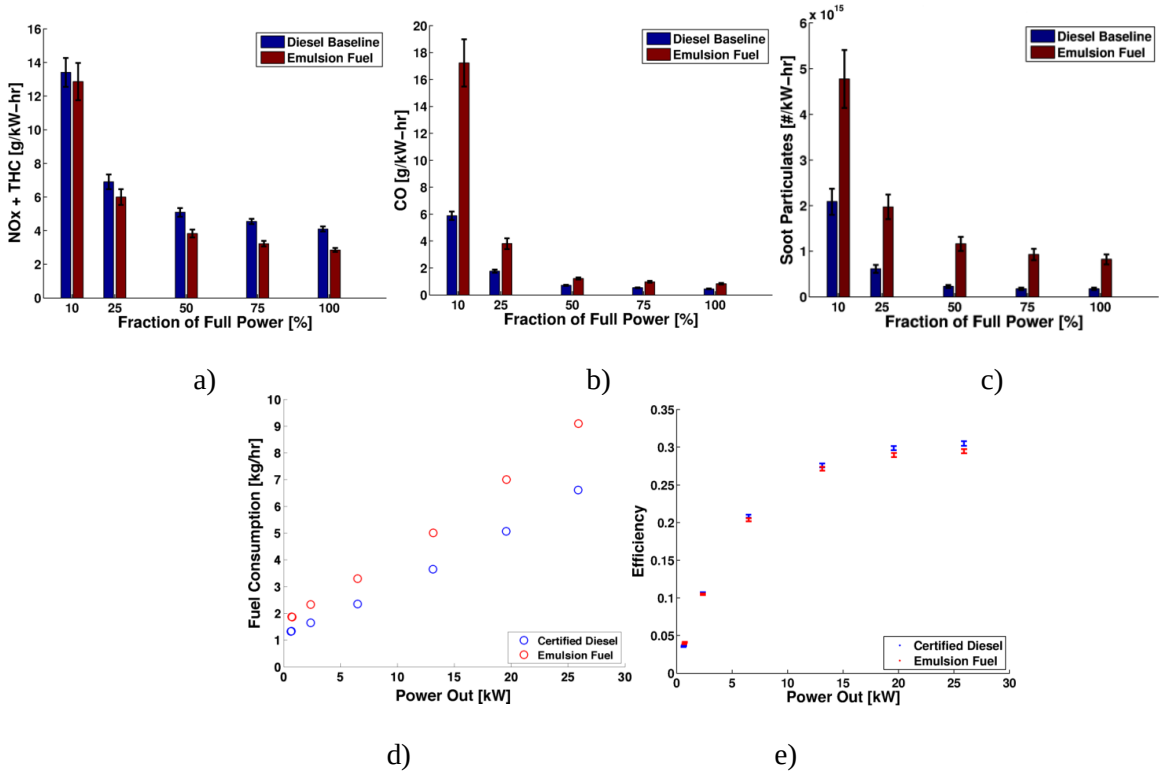


Figure 3: Emissions and fuel performance from certified diesel and glycerol/water/diesel emulsion fuel testing on a CAT C2.2 marine diesel generator over an ISO 8178 duty cycle. Energy weighted mass emissions of a) NO_x +THC and b) CO, energy weighted number emissions of c) soot particulates, d) fuel consumption as a function of power output, and e) fuel efficiency as a function of power output averaged over each load setting.

Additional testing conducted on board the research vessel *Quickwater* was found to be significantly different than results observed in the indirect injection diesel generator. Figure 4 illustrates gaseous and particulate emissions collected on board *Quickwater* for diesel and the glycerol/diesel emulsion fuel. The maximum load achievable with the emulsion fuel was approximately 87.5% of the maximum engine rating for diesel, as reflected in Figure 4, and attributed to the lower energy density of the emulsion fuel and a miss-matched engine fuel pump for the application. Figure 4a illustrates emissions of NO_x + THC remained unchanged over all load settings for the two fuels. Figure 4b shows CO emissions for the emulsion fuel were elevated at 25% load and reduced at all other load settings. In contrast, soot particulate number counts shown in Figure 4c are shown consistently elevated over the diesel baseline. Fuel consumption as a function of generator output power is illustrated in Figure 4d. Fuel consumption is again shown as elevated for the emulsion fuel due to the lower heating value of the fuel. Thermodynamic efficiency exhibits little dependence on fuel type as shown in Figure 4e.

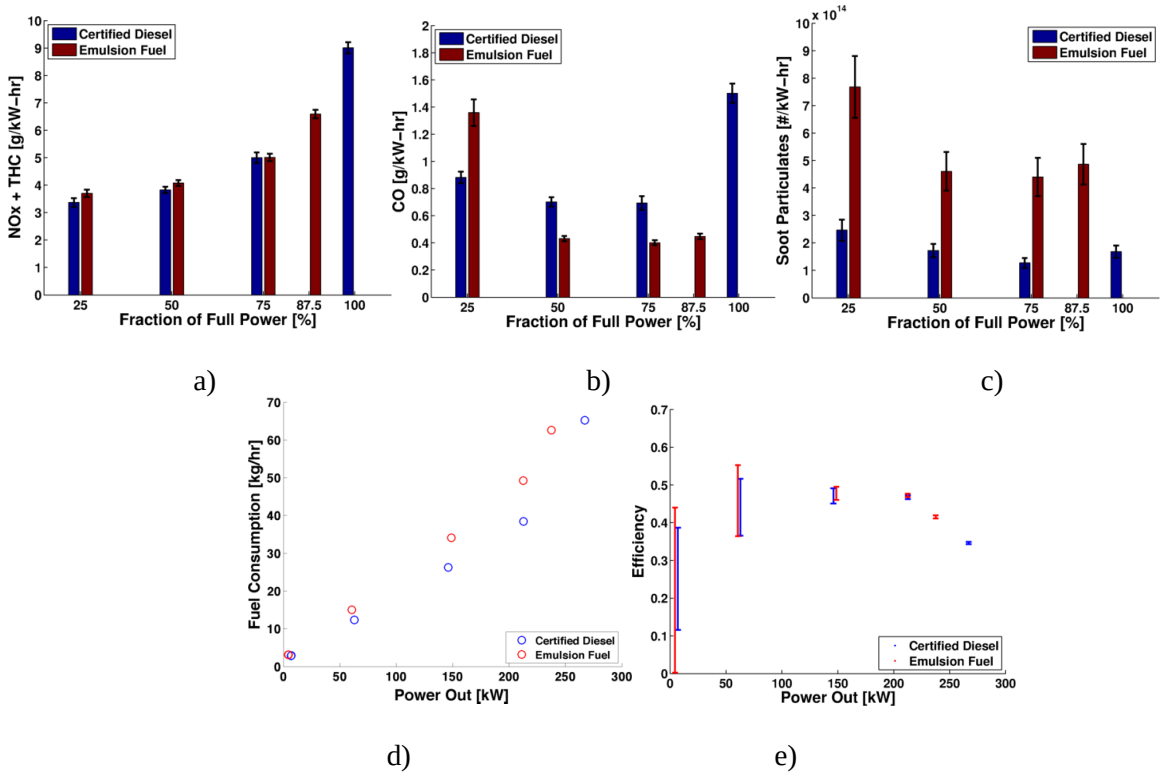
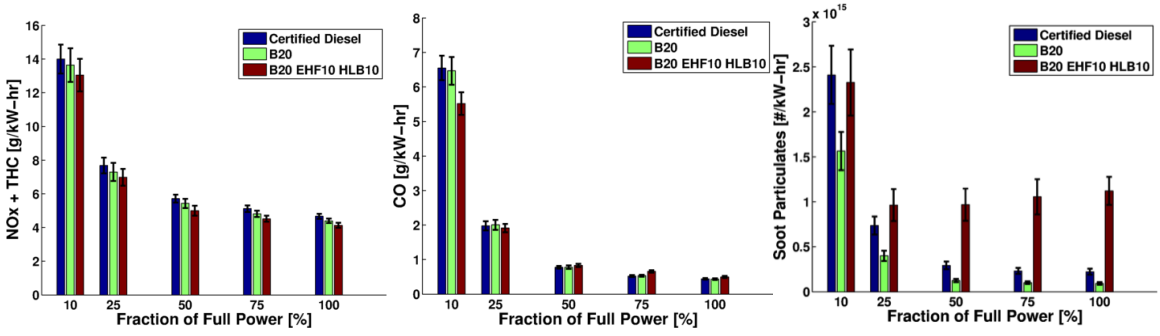


Figure 4: Emissions from certified diesel and glycerol/water/diesel emulsion fuel testing on the research vessel Quickwater over an ISO 8178 duty cycle. Energy weighted mass emissions of a) NO_x +THC and b) CO, energy weighted number emissions of c) soot particulates, d) fuel consumption as a function of power output, and e) fuel efficiency as a function of power output averaged over each load setting. Note the large error bars for fuel efficiency at low loads are due to entrained air in the fuel return line introducing considerable noise to the fuel flow measurement.

The persistent differences in soot number concentration prompted additional testing in the laboratory on the CAT C2.2 test cell. Additional tests were conducted via gravimetric soot sampling to determine total soot mass and thermophoretic soot sampling with transmission electron microscopy image analysis to determine particle size. The results indicate the emulsion fuel produced soot particulates of a smaller mean particle size with total soot mass equal to the diesel baseline fuel. The reduction in particulate size is consistent with other results reported in literature for oxygenated fuels with the cause likely due to a reduction in flame equivalence ratio and a suppression of soot precursor species [7,8]. However, the effect of decreasing particle size is typically associated with a reduction in total soot mass. With no evidence of a reduction in total soot mass, other additional effects may also play a role. Changes in fuel atomization due to variations in fuel viscosity and surface tension is a likely candidate that deserves further attention.

4.2 Glycerol/B20 Biodiesel Emulsion

The evolution of emulsion fuel development continued with an exploration of biodiesel blends. An experimental emulsion fuel blend of 8% glycerol, 2% water, 85% B20 biodiesel (20% biodiesel, 80% 2007 Cert. diesel (vol/vol)), 2.7% Tween 80 and 2.3% Span 80 by weight was chosen for optimum fuel stability and tested on the CAT C2.2 laboratory test cell using the procedures described above. The biodiesel was sourced from a local waste vegetable oil biodiesel producer. The biodiesel was produced batch-wise using base-catalyzed transesterification. The resulting fuel is refered to as B20EHF10 denoting the B20 base-fuel use and the 10 wt% emulsion in the mixture. It should be noted that the resulting HLB value of 10 used in the biodiesel containing fuels are the same HLB values used in ULSD only emulsion stabilization. We confirmed that stabilized emulsions can be made using this surfactant formulation with the stipulation that the relative amount be increased from 3 wt% (ULS-only case) to 5 wt% (B20 case). Figure 5 illustrates the gaseous and particulate emissions measured for diesel, B20 biodiesel, and the glycerol/water/B20 biodiesel emulsion fuel. A modest reduction in energy weighted NOx + THC emissions is observed in Figure 5a for the B20 biodiesel and biodiesel emulsion fuel in comparison to diesel with the emulsion fuel exhibiting the lowest NO_x + THC emissions. Energy normalized CO emissions shown in Figure 5b were unchanged for diesel and B20 biodiesel within uncertainty bounds. CO emissions for the B20 biodiesel emulsion fuel were slightly reduced at 10% load and slightly elevated at higher loads. Figure 5c depicts energy weighted particulate number emissions. Particulate counts are shown reduced on average for the B20 biodiesel in comparison to diesel and elevated on average for the B20 biodiesel emulsion fuel in comparison to the other two test fuels.

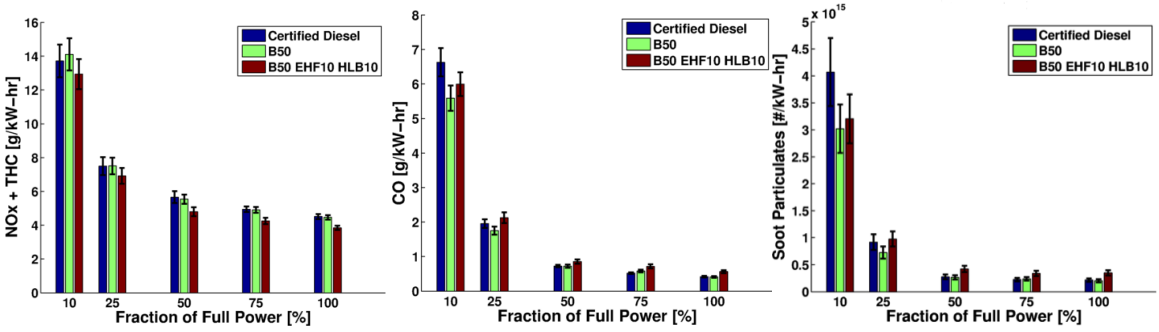


a) b) c)

Figure 5: Emissions from certified diesel and glycerol/water/B20 biodiesel emulsion fuel testing on a CAT C2.2 marine diesel generator over an ISO 8178 duty cycle. Energy weighted mass emissions of a) NO_x +THC and b) CO, and energy weighted number emissions of c) soot particulates averaged over each load setting.

4.3 Glycerol/B50 Biodiesel Emulsion

Lastly, an experimental emulsion fuel blend of 8% glycerol, 2% water, 85% B50 biodiesel (50% biodiesel, 50% 2007 Cert. diesel), 2.7% Tween 80 and 2.3% Span 80 by weight was explored and tested on the CAT C2.2 laboratory test cell. Again, the resulting fuel was stabilized using the HLB 10 surfactant formulation as indicated above in B20 testing fuel composition. The resulting fuel is referred to as B50EHF10 denoting the use of B50 base-fuel and that the fuel is comprised of 10 wt.% emulsion. Figure 6 illustrates the gaseous and particulate emissions measured for diesel, B50 biodiesel, and the glycerol/water/B50 biodiesel emulsion fuel. A modest reduction in energy weighted NO_x + THC emissions is observed in Figure 6a for the biodiesel emulsion fuel with the diesel and B50 biodiesel exhibiting equal NO_x + THC emissions. Energy normalized CO emissions shown in Figure 6b were unchanged for diesel and B50 biodiesel within uncertainty bounds. CO emissions for the B50 biodiesel emulsion fuel were slightly reduced at 10% load and slightly elevated at higher loads. Figure 6c depicts energy weighted particulate number emissions. Particulate counts are shown reduced on average for the B50 biodiesel in comparison to diesel with near parity observed between the diesel and B50 biodiesel emulsion fuel within uncertainty bounds. The detailed chemical and physical mechanisms driving changes in particulate emissions are currently unknown. It is postulated that changes in chemical pathways along with changes in fuel atomization likely play a role and deserve further study.



a) b) c)

Figure 6: Emissions from certified diesel and glycerol/water/B50 biodiesel emulsion fuel testing on a CAT C2.2 marine diesel generator over an ISO 8178 duty cycle. Energy weighted mass emissions of a) NO_x +THC and b) CO, and energy weighted number emissions of c) soot particulates averaged over each load setting.

5. Conclusions and Future Work

The activities of METEL, at Maine Maritime Academy, in the area of alternative emulsion fuel development and testing were presented. An overview of METEL is discussed including its unique capabilities in the fabrication, characterization and engine testing of emulsion fuels. Glycerol is an attractive fuel feedstock as it is an inexpensive waste product of biodiesel production with favorable combustion

characteristics. The emissions and performance of glycerol/diesel and glycerol/biodiesel emulsion blends on marine diesel engines in the laboratory and on the research vessel *Quickwater* are presented. Fuel characteristics and handling issues for these fuels are discussed.

Biofuel emulsions are a unique alternative fuel concept with demonstrated application to maritime operations. While considerable work in literature has focused on characterizing the combustion and performance of popular biofuel blends, the detailed chemical and physical mechanisms driving changes in NO_x, THC, CO, and particulate emissions for biofuel emulsions remain an open area of investigation. It is postulated that changes in chemical pathways along with changes in fuel atomization likely play a role and deserve further study.

Acknowledgements

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