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**A GLOBAL SURVEY OF THE INCIDENCE OF FAME AND MICROBIAL CONTAMINATION IN MARINE DISTILLATE FUELS**

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**ABSTRACT**

On 1st January 2015 the sulphur content of marine fuels in use inside Emission Control Areas (ECA) reduced from 1.00% to 0.10%. Ultra low sulphur automotive diesel which contains Fatty Acid Methyl Esters (FAME) at up to 7%, are already widely used in Europe, USA and other global regions. Given the complex supply chain of marine bunker fuels and the increasing demand for low sulphur gas oil it would seem inevitable some fuel blended with biodiesel will find its way into the marine fuels supply chain. To evaluate the true extent of FAME (biodiesel) contamination in the marine distillate fuels, a joint survey was conducted by Lloyds Register FOBAS, Guardian Marine Testing Ltd (GMT) and ECHA Microbiology Ltd. Nearly 2400 samples of marine distillate fuel derived from wide range of ports, suppliers & barges and considered to represent a good overview of the global market for marine distillates, were tested for compliance with ISO 8217 standard parameters and additionally for FAME contamination, using IR spectroscopy. 116 (4.68%) of the samples were positively identified with FAME and were then tested by the reference EN 14078 method to accurately quantify the level of FAME contamination. All samples which were identified with FAME, along with 32 randomly selected FAME-free samples, were then analysed for viable microbial content (IP 613) and water content by Karl Fisher analysis (ASTM D6304). A strong correlation between viable microbial contamination and water content was observed. Marine distillate fuel containing FAME was found to be more likely to contain viable microorganisms. Marine fuel containing FAME is more likely to contain a higher level of viable contamination than marine fuel lacking FAME.

**KEYWORDS**

Microbial contamination, Fatty Acid Methyl Esters, FAME, marine distillate fuels, biodiesel blends, Emission Control Areas, ECA, low sulphur, ISO 8217:2012, EN14078:2009, IP613/14, ASTM D7978-14, ASTM D6304-07.

**INTRODUCTION**

The more widespread use of Fatty Acid Methyl Esters (FAME) in automotive diesels (biodiesel blends) and burner fuels has seen a dramatic increase in incidents caused by microbial contamination in recent years. Biodiesel is much more readily degraded by microorganisms than mineral diesel and fuels containing biodiesels are prone to faster and more extensive microbial spoilage. A study by the Energy Institute showed that water content in diesel increased with increasing FAME concentration.1 It is well documented that water is a key influence on the susceptibility of a fuel system to microbial growth. The study showed FAME-free diesels were notably less prone to microbial contamination. Zero sulphur automotive diesel containing ≥2% FAME was significantly more susceptible to microbial growth and the amount of microbial contamination and microbial biomass increased with increasing FAME concentration. Biodiesels have a hygroscopic affinity to absorb water into the fuel, either dissolved or as suspended as micro-droplets, and this may directly stimulate microbial growth2 and also make existing water control procedures less effective.3 Microbial growth in fuel systems can lead to serious operational problems such as fuel filter clogging, injector malfunction and corrosion.4 Other fuel quality issues associated with biodiesels blends include;5,6

* Poor oxidation stability leading to long term storage issues.
* Presence or formation of insoluble particulates (e.g. glycerides and sterol glucosides).
* Difficulties in maintaining homogenous blends.
* Poor flow characteristics when at low temperatures (waxing).
* An increase in the total acid number (TAN) of the fuel.
* Corrosivity to certain materials (e.g. rubber gaskets, hoses & seals may swell).
* Adherence of FAME to exposed surfaces (metal & glass) including filter elements.

Diesel containing FAME is already widely used in Europe, USA and other global regions. Ultra low sulphur automotive diesel (e.g. ASTM D975 and EN 590), and other middle distillate fuels such as burner fuels intended for non-marine use (e.g. BS 2869), generally allow 5 to 7% FAME. Because fuel supply chains cannot provide complete segregation of fuel types in distribution, there is a potential for small amounts of FAME to be introduced to marine fuels by cross contamination, for example in multiproduct pipelines. This has already been shown to be a concern for aviation fuels.7 Additionally, because in many respects non-marine diesel fuels meet the requirements for marine use, there is a concern where they are supplied for marine applications. This has been considered in some marine fuel specifications, such as ISO 8217:2012 for Marine DMA, which currently only allows a *de minimis* concentration of up to 0.1% FAME by volume8, although there are proposals for this limit to be relaxed for DMA to 0.5%.9 There are also proposals for specific grades of marine distillate (DFA, DFZ and DFB) which include FAME at up to 7 %.

On 1st January 2015 the sulphur content of marine fuels in use in Emission Control Areas (ECA) reduced from 1.00% to 0.10%. 10. The ECAs cover a wide global region including the Baltic Sea, the North Sea, designated coastal areas off the United States and Canada and the Caribbean Sea area around Puerto Rico and the US Virgin Islands. Given the complex supply chain and increasing demand for ultra low sulphur marine distillate it seems probable that some fuels containing FAME concentrations >0.1% will find their way into the marine supply chain.

With increasing industry awareness, quality issues with automotive and non-marine diesels blended with FAME can usually be managed by good husbandry. Due to the relatively high turnover in distribution and use, progressive fuel deterioration due to microbial growth and poor oxidation stability will usually have minimal impact. Typically automotive diesels are burned within a matter of weeks of production and usually there is no prolonged storage. However, marine distillate fuels may be stored on-board for prolonged periods. For example, ultra low sulphur marine distillate can be bunkered and then stored on-board until needed by the vessel when it is in an ECA. There is thus more time and scope for deterioration and fuel quality issues when FAME is present in marine distillate and ultimately this could lead to increased operational problems. This will be particularly so where the user is unaware that the fuel they have purchased contains FAME and they are unable to take appropriate precautionary measures such as those described by CIMAC (the International Council on Combustion Engines).11

**INVESTIGATION**

To evaluate the true extent of FAME contamination in marine distillate fuels, a total of 2346 samples were obtained from a wide range of ports, suppliers and barges over a 3 month period in 2013. The samples were considered to provide a good representation of the global bunker supply market for marine distillate. Although marine distillate fuels would generally be supplied against the ISO 8217:2012 DMA specification, it is not known which specification was relevant to the supply of all the fuel represented by the samples tested. Nevertheless, it is understood that in the majority of cases, the fuel would be expected to comply with the ISO 8217:2012 requirement for no more than the *de minimis* limit 0.1% FAME.

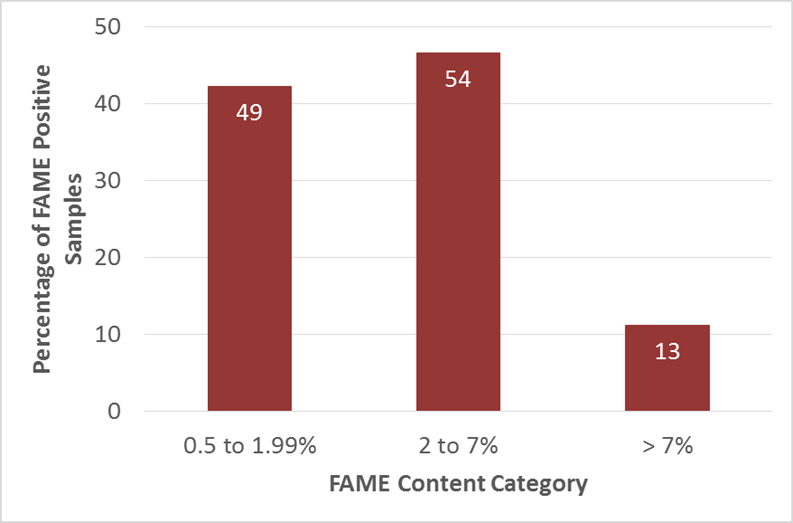
All samples were tested for compliance with the ISO 8217:2012 DMA specification and additionally screened for the presence of FAME using an in-house FTIR method which had a FAME detection limit of 0.5% v/v. This method is essentially a “Go / no-go” method which can be used to routinely screen samples for FAME quickly and cost efficiently. Those samples which contained >0.5% FAME, denoted as “FAME positive”, were subsequently tested using the reference EN 14078:2009 infra-red spectroscopy method to accurately quantify the level of FAME contamination. This is the method specified in the ISO 8217 specification. The FAME positive samples along with 32 randomly selected “FAME negative” samples (i.e. those found to contain <0.5% FAME by in-house FTIR method) were also tested for viable microbial content by IP 613/14 (ASTM D7978-14) and for water content by Karl Fischer analysis (ASTM D6304-07).

**RESULTS**

**FAME content**

IR spectroscopy showed that 5% of all samples tested contained >0.5% v/v FAME. After subsequent tests to accurately quantify FAME by EN 14078:2009 method, samples were categorised into three groups according to FAME content (see Figure 1). Most of the FAME positive samples contained 2 to 7% v/v FAME. More than 7% v/v FAME was detected in 13 samples; the highest concentration of FAME detected in all samples was 57% v/v. The FAME contamination varied by geographical region, but was found to be most prevalent in Asia, Southern Europe and Western Africa.

*Figure 1. Fame content in FAME positive samples.*

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**The incidence of FAME and microbial contamination**

45% of the FAME positive samples also contained microbial contamination. It should be noted that, due to the global nature of the survey, it was not possible to ensure that samples were tested within 24 hours of them being taken, which is generally considered best practice. However, in our opinion delays between sampling and testing would, if anything, cause a reduction of detected microbial contamination rather than an increase. Thus the survey may underestimate the incidence and levels of microbial contamination relative to condition at time of bunkering.

To help understand the relationship between the presence of FAME and microbial contamination levels, FAME positive and FAME negative samples were categorised into five microbial contamination levels depending on the number of microbial Colony Forming Units (cfu) detected by the IP613 test. Because a 0.25 ml volume of fuel sample is tested by the IP 613 method, contamination categories can be defined in increments of 4000 cfu/L (because each colony in the test equates to 4000 cfu/L).

|  |  |
| --- | --- |
| **Microbial Contamination Category cfu/L (IP 613/14)** | |
| < 4000 (Not detected) | No contamination |
| 4000 | Light contamination |
| >4000 <20,000 | Moderate contamination |
| 20,000 – 1,000,000 | Heavy contamination |
| >1,000,000 | Very heavy contamination |

It should be noted, that the above limits are not globally accepted standards for marine diesel and no such standard specifications exist. Whilst we would expect most marine distillate fuels to contain <4000 cfu/L, it is not unusual for diesel to contain between 4000 and 20,000 cfu/L. We would typically consider contamination in excess of 20,000 cfu/L in a representative fuel sample to be above normal and indicative of microbial growth in the tank or system sampled. A contamination in excess of 20,000 cfu/L would be considered heavy, although operational problems might only be experienced when the contamination is in excess of 1,000,000 cfu/L. There are no hard and fast correlations between fuel fitness for use and detected microbial contamination level (cfu/L) and other test data and information should be considered.

The level of microbial contamination for FAME positive and FAME negative samples are shown Figure 2. The results indicate that FAME positive samples were more likely to contain viable microorganisms. Furthermore, FAME positive samples were more likely to contain higher levels of viable microbial contamination, for example in the moderate, heavy or very heavy contamination categories, than the FAME negative samples.

*Figure 2. The level of microbial contamination in FAME positive and FAME negative samples.*



**Investigation of the relationship between measured FAME content, water content and microbial contamination.**

Samples were categorised into groups by water content (0 to 25 ppm m/m, 25 to 50 ppm mm and so on in 25 ppm iterations). The average water content in each of these groups was plotted against average FAME content (Figure 3) and average microbial content (Figure 4). The number of samples in each water content category is also shown. A correlation was found between increasing FAME and water content; the higher the FAME content, the higher the water content. There was a strong correlation between the numbers of viable microbes detected and water content; the higher the water content, the higher the viable microbe count.

*Figure 3. Average water content plotted against average FAME content.*



*Figure 4. Average water content plotted against average numbers of microbes detected.*



When comparing the full range of FAME concentrations detected with the microbial content data, there was no direct correlation between the numbers of viable microorganisms and FAME concentration. However, a correlation was found in samples containing between 0 and 2% FAME as shown in Figure 5; the higher the FAME content, the higher the viable microbe count. This finding concurs with the Energy Institute study previously discussed which indicated that it is somewhere between the range 0.4% and 2% FAME that fuel becomes significantly more susceptible to microbial growth.

*Figure 5. Average FAME content plotted against average numbers of microbes detected.*



**DISCUSSION**

The results of the joint study indicate that, on current trends, the risk of receiving FAME and/or microbial contamination should be taken seriously. Depending on trading patterns, some vessels are more susceptible to the supply of contaminated fuels than others. The changes to sulphur regulation in Emission Control Areas (ECAs) will increase the demand for zero sulphur marine distillates and are expected to affect the quality of fuels available for supply in these regions. It will likely result in the increased supply of fuel meeting distillate specifications which allow FAME at concentrations up to 7%. Where users are aware of this they can take various precautions such as those described in the CIMAC *Guidelines for Ship owners and Operators on Managing Marine Distillate Fuels Containing up to 7.0% v/v FAME (biodiesel)*.11 These stipulate good practice in respect of fuel storage, turnover and testing.

Conversely, where marine fuel users are not aware of the presence of FAME in distillate fuel they purchase, they may find that fuel husbandry measures historically adopted may no longer be sufficient for control of microbial growth and other quality issues. If significant concentrations of FAME are present, even if distillate is supplied on specification, it may become unfit for use where there is low fuel turnover, long term storage or a significant risk of water ingress. Users would be advised to bunker from fuel suppliers who can offer assurances that the distillate they supply for marine use will not contain more than *de minimis* concentrations of FAME, even though higher concentrations may be allowed in the specification.

**SUMMARY**

A global survey of marine distillate fuels found that;

* 5% of 2346 samples from a wide range of ports, suppliers and barges were FAME positive, i.e. they contained more than 0.5% v/v FAME.
* Most of the FAME positive samples contained 2 to 7% v/v FAME.
* The highest concentration of FAME detected in all samples was 57% v/v.
* The FAME contamination varied by geographical region, but was found to be most prevalent in Asia, Southern Europe and Western Africa.
* Of the FAME positive samples, 45% contained some level of microbial contamination.
* FAME positive samples were more likely to contain viable microorganisms and were more likely to contain significant levels of viable microbial contamination (above 4000 cfu/L) than the FAME negative samples.
* Within the range 0 to 2% v/v FAME, the level of microbial contamination increased with increasing FAME content. There was no clear correlation between FAME concentration and level of viable microbial content in samples which contained above 2% v/v FAME.
* A correlation was found between increasing FAME and water content; the higher the FAME content, the higher the water content.
* There was a strong correlation between the numbers of viable microbes detected and water content; the higher the water content, the higher the viable microbe count.

The results from the joint study indicate that, on current trends, the risk of receiving FAME and/or microbial contamination should be taken seriously.

**REFERENCES**

[1] EI Research Report: Investigation of microbiological susceptibility of biodiesel and biodiesel blends, 2014.

[2] E. C. Hill & G. C. Hill, Strategies for resolving problems caused by microbial growth in terminals and retail sites handling biodiesels in *Proceedings of the 11th International Conference on Stability, Handling and Use of Liquid Fuels*, IASH, Prague 2009.

[3] EI Microbiology technical bulletin sheet 1: Microbial growth in biodiesels and other fuels containing fatty acid methyl esters (FAME). (May 2011)

[4] IP Guidelines for the Investigation of the Microbial Content of Petroleum Fuels and for the Implementation of Avoidance and Remedial Strategies, 2nd Edition, Energy Institute (IP Publications), 2006

[5] P. Livingston, G. Hill & N. Javaid, Fuel Quality – Close Analysis in *Bunkerspot*, Aug/Sept 2014

[6] Implications of Biofuels on Microbial Spoilage and Corrosion within the Fuel Distribution Chain and End Use, Literature Review by A. Price, (2008), Energy Institute, London

[7] V. Hughes & A. Kitson-Smith, The impact of the introduction of biodiesel on jet fuel distribution and quality in *Proceedings of the 11th International Conference on Stability, Handling and Use of Liquid Fuels*, IASH, Prague 2009.

[8] ISO 8217:2012 Petroleum products - Fuels (class F) - Specifications of marine fuels, 5th Edition, 15-08-2012

[9] ISO/TC 28/SC 4 N 887, ISO 8217:2012 Petroleum products - Fuels (class F) - Specifications of marine fuels, ISO/TC 28/SC 4/WG 6 draft, 01-12-2014

[10] International Convention for the Prevention of Pollution form ships (MARPOL) Annex VI (Regulations for the Prevention of Air Pollution from Ships), 2008

[11] CIMAC Working Group 7 – Fuels (WG7) Guidelines for Ship owners and Operators on Managing Marine Distillate Fuels Containing up to 7.0% v/v FAME (biodiesel), V1.0, 2013.