Comparative Study of Light Cycle Oil and Naphthalene as an Adequate Additive to Improve the Stability of Marine Fuels

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ABSTRACT: Post the enforcement of the Global Sulphur Cap 2020, large amounts of marine fuel have been produced as a mixture of two or more components to achieve a sulfur level of less than 0.5 wt %. This has led to the wider use of diluents or cutter stocks from different origins to comply with the quality requirements of the ISO 8217 standard. These can often be bio- or alternative components with a lower level of stability. This makes it crucial to select components that are compatible and form a homogeneous mixture. The ISO 8217 standard provides commercial and operational protection; however, the stability of the fuel deteriorates over time. Therefore, the purpose of this study was to develop an adequate component or additive to improve the colloidal stability of marine fuels. A set of laboratory-prepared samples was used to verify the positive effects of the addition of a light cycle oil and also a newly developed additive (containing 30 wt % naphthalene as the simple diaromatic hydrocarbon in tetralin). The prepared fuels (with effective additive dosage), when stored for 60 days, maintained the total sediment accelerated (TSA) parameters, confirming the stability of the fuels. The additive converted the unsaleable fuel into fuel that complied with the requirements of ISO 8217. The developed additive can be used for industrial applications in marine fuel production.

1. INTRODUCTION

High-viscosity residual marine fuels are produced as a multicomponent mixture. They predominantly contain aromatic polar molecules with high-molecular-weight asphaltenes.1 The asphaltenes remain in a colloidal dispersion with the surrounding maltenes, as long as the solubility force (dispersion, polarity, and hydrogen bonding) is in effect.2 The solubility of asphaltenes is a key indicator of colloidal stability;3 it is reduced by the addition of nonpolar solvents, including distillates, which can lead to overall instability.2 Marine fuel producers and their end-users face problems associated with fuel instability, which causes sludge formation in storage tanks and plugging of marine engine systems.4 This instability is caused by repeated thermal stress or aging during long-term storage.5 The free radicals and aromatic hydrogen contents in the residues from visbreaking (thermal cracking) decrease with the increase in storage time.6 Therefore, marine fuel should be produced with a certain degree of aromaticity reserve to prevent the precipitation of asphaltene molecules.6

The quality of the final residual marine fuel, produced by mixing residual fractions and cutter stock, is determined by the stability and sediment content in the unconverted residual fraction.7 However, appropriate selection of the cutter stock also plays an important role.7,8 The characteristics and basic properties of a suitable cutter stock have acquired considerable significance post the implementation of IMO 2020 for reducing the sulfur content to less than 0.5 wt %. The amount of cutter stock needed to reach the required viscosity and sulfur content may be more than 30 wt %.5,9,10 Moreover, the entire mixture must be compatible, i.e., it should not present material separation, which could result in the formation of multiple phases (asphaltene flocculation and precipitation).

The stability must be guaranteed throughout the production and supply chain by means of the parameter of total sediment aged according to ISO 10307-2.11 The maximum value of this parameter (0.1 wt %) is specified in the ISO 8217 standard.12 Two aging procedures are available for this purpose: thermal (TSP = total sediment potential) and chemical (TSA = total sediment accelerated). The reference test method according to ISO 8217 is the TSP method; however, in practice, the TSA method is preferred owing to its lower time consumption.
Other recommended methods\(^1\) for assessing the stability or compatibility of marine fuels are the \(S\)-value,\(^{13-15}\) \(P\)-value,\(^{16}\) and \(P\)-ratio.\(^{17}\) The aforementioned methods are based on dissolving the sample and subsequently titrating it with an appropriate agent. In some cases, the \(S\)-value does not correlate with the total sediment content, i.e., with the colloidal stability of the residual marine fuel, especially for fuel based on H-oil residual fractions.\(^3\) Additionally, these methods require expensive, single-purpose laboratory equipment. In contrast, the microscopic analysis offers a good correlation with the colloidal stability and compatibility of marine fuels.\(^{18-20}\) Furthermore, a spot test based on ASTM D4740\(^{21}\) is frequently used as a rapid and simple method for this type of assessment.\(^2,4,22\)

The addition of suitable substances or reagents is one possible way to improve fuel quality. Accordingly, commercially available dispersants, cold-flow improvers, lubricity improvers (lubricant for the reduction in friction and or wear the mechanical parts of engine), cetane number (indicator of the combustion speed of marine fuel and compression needed for ignition) improvers, and combustion catalysts are widely employed in the shipping sector.\(^23\) Currently, it is necessary to use lubricity improvers to reduce the sulfur content in marine fuels.\(^{24}\) In partial switching to distillate-type marine fuels, adjustment of the cold-flow properties by depressant-dispersing additives has been frequently discussed.\(^{9,10,25}\) Additionally, many studies have focused on improving the emission characteristics and their measurement methods.\(^{23,26,27}\)

Unfortunately, only a few studies have focused on the additivation of marine fuel that does not meet the requirements for the content of total sediments according to ISO 8217. Mitusova\(^4\) reported that the fuel stability can be improved by additivation (targeted improvement of selected properties) to keep the asphaltene-resinous substances in a suspension. The additives based on hydroxyethylated amines (Bunkersol D, Amerod) and alkyl naphthalenes (VNII NP 200, Russia) as the active agent were tested. However, these additives are ineffective if the marine fuel contains a large number of unstable components.\(^4\) The use of these additives must be taken into account already during production; according to the presented data, it is not possible to modify the already produced unsuitable (unstable) material with their help. In such cases, it is necessary to identify an adequate alternative solution.

The purpose of this study was to identify an additive that would maintain the stability parameter within the required limit, even for marine fuels containing unstable components, such as a visbreaking residue. According to the available information, this type of research has not been published anywhere yet. The total sediment contents after chemical aging, spot testing, and microscopic analysis were used to verify the effectiveness of the newly designed additive. Additionally, we performed a complete evaluation of the
properties, including the storage stability of the prepared marine fuel, with the new additive.

2. RESULTS

In the content analysis of saturates, aromatics, resins, and asphaltenes (SARA) (Figure 1), the aromatics (the largest group) exhibited a double-peak signal for a vacuum residue from the visbreaking unit (VR VBU). The last peak of the saturated group represented the smallest area according to the IP 469 method.

A comparison of atmospheric gas oil from the visbreaking unit (AGO VBU) with light cycle oil (LCO) showed significant differences in the content of individual aromatics, based on the results of high-performance liquid chromatography (HPLC) (Table 3) and two-dimensional gas chromatography with time-of-flight mass spectrometry (GC × GC-TOFMS) (Figures 2 and 3). AGO is mostly monoaromatic, whereas LCO is derived from diaromatic hydrocarbons.

The distillation curves (Figure 4) of the individual streams were consistent with the observations from a comparison of...
The production of low-sulfur marine fuels. This posed a limitation in the group of diaromatic hydrocarbons. This posed a limitation in the group of diaromatic hydrocarbons. This posed a limitation in the group of diaromatic hydrocarbons. This posed a limitation in the group of diaromatic hydrocarbons. This posed a limitation in the group of diaromatic hydrocarbons.

The base-case marine fuel sample was prepared at the laboratory scale by mixing 70 wt % of VR VBU with 30 wt % of AGO VBU. The marine fuel met the density and viscosity requirements for the ISO-F-RMK-700 category according to ISO 8217. However, the TSA parameter was evaluated to be unsatisfactory because it was undetectable by hot filtration. The sludge formed during the TSA procedure after the addition of n-hexadecane caused plugging of the glass filters. A solvent mixture (heptane–toluene, 85:15 vol %) was added to the sample during hot filtration. This procedure conflicted with the procedure laid down in the ISO 10307 standard; therefore, the results were only indicative. Hence, we investigated the possibility of improving the colloidal stability and further reducing the key qualitative parameter of the TSA.

2.1. LCO Addition. The LCO was tested as a suitable cutter stock to improve the colloidal stability. According to the analyses, it contained a relatively large amount of diaromatics (>70 wt %), which resulted in the stabilization of asphaltenic macromolecules in the colloidal suspension. Four samples of marine fuels were prepared with different LCO contents (Figure 5). Subsequently, the TSA parameter was determined to investigate the effects of LCO addition.

2.2. Additivation. The use of pure hydrocarbons would solve the problem of high sulfur content and reduce the effective dose. The majority of the LCO consists of the diaromatic fraction (87.1 rel % according to GC × GC-TOFMS). Because of the positive effect of LCO addition, the effect of naphthalene (the simplest pure diaromatic hydrocarbon consisting of a fused pair of benzene rings) was also investigated. This approach could be economically advantageous for refinery–petrochemical complexes that contain naphthalene in their standard product portfolio. The positive effect of substituted naphthalenes has been studied in the past. Naphthalene is a crystalline solid with a melting point of 80.3 °C; this melting point considerably complicates the easy application and subsequent homogenization of marine fuels. The temperature for the laboratory preparation (blending and subsequent homogenization) of marine fuels does not exceed 80 °C because there is a risk of loss of light components or unwanted aging at higher temperatures. Additionally, it is necessary to consider that a part of naphthalene transforms into its crystalline form during marine fuel cooling. These challenges make the use of pure naphthalene difficult at both the laboratory and industrial scales.

Therefore, a suitable solvent was required to provide naphthalene in the form of a liquid, easy-to-apply additive. A partially hydrogenated derivative of naphthalene, i.e., 1,2,3,4-tetrahydronaphthalene (tetralin), was used as a solvent. Tetralin is a liquid substance with a melting point of −35.8 °C. The additive was prepared at the laboratory scale by mixing 30 wt % naphthalene and 70 wt % tetralin. When the tetralin content was less than 70 wt %, naphthalene was recrystallized within 2 days. A 30 wt % solution of naphthalene was successfully tested as a stable additive in a permanently liquid form under laboratory and normal process conditions. Seven samples of marine fuels with varying concentrations of this additive (0.01−5 wt %) were prepared to monitor their effectiveness (Table 1).

Table 1. Summary of TSA Assessment in Marine Fuel with the Additive

| additive content (wt %) | 0 | 0.01 | 0.1 | 1.0 | 1.5 | 2.0 | 3.0 | 5.0 |
|------------------------|---|------|-----|-----|-----|-----|-----|-----|
| naphthalene content (wt %) | 0 | 0.003 | 0.03 | 0.3 | 0.45 | 0.6 | 0.9 | 1.5 |
| TSA (wt %) | undetectable | 7.30a | 5.19a | 3.44a | 0.08 | 0.07 | 0.06 | 0.06 |

aValue should be reported as >0.5 wt % according to ISO 10307.

Figure 6. Marine fuels containing the additive in a low concentration (unstable samples).
were not apparent. Additionally, a significant difference between the unstable (Figure 6) and stable (Figure 7) samples of marine fuels was evident in a visual comparison of the separated sediments on glass filters.

A visual comparison of lower filters (the rear ones in Figures 6 and 7), which were darker for stable samples, indicated a higher homogeneity in the oil phase. This confirmed the positive effect of the additive on the overall stabilization of the colloidal structure.

The selected samples (from Table 1) were evaluated in more detail by microscopic analysis and calculations of the \( \nu \)-value parameter to obtain complementary information about the colloidal stability. For this purpose, we compared the original marine fuel, the marine fuel (from the group of unstable samples) containing 0.1 wt % of the additive, and the marine fuel meeting the standardized requirements, containing 2 wt % of the additive. Changes in the structure of the selected marine fuels were evident from a comparison of their microscopic images (Figure 8).

As a result of additivation, the number and size of the visible clusters, which were based on the asphalteneic macromolecules, decreased. The middle diameter of the clusters decreased from \( \sim 20 \) to \( \sim 10 \) \( \mu \)m. More importantly, the reduction in the total particle number and the reduction in the ability to form larger clusters inhibited the plugging of glass filters. The glass microfiber filters used for the TSA assessment were made of borosilicate with a pore size of 1.6 \( \mu \)m (GF/A grade) according to ISO 10307-2. As shown in Figure 6, the plugging of the filters and the subsequent formation of a layer of the material caused difficulty in the passing of the sample through the filter and resulted in very high TSA values. Therefore, the tendency to form large clusters was evaluated using LAS X software (Leica Microsystems, Germany) (Figure 9).

The mapping of the clusters exhibited significant differences between the individual samples in relation to the contents of the additive. The largest (most representative) size group is marked in red. This group constituted >95, 88, and \( \sim 83\% \) of the total number of clusters in the samples without the additive, with the additive at 0.1 wt %, and with the additive at 2 wt %, respectively. The susceptibility to the formation of larger clusters was evident for the base-case sample without additives. The total number of clusters and their total areas decreased because of the additive effect (Figure 10).

The total number of clusters was reduced by more than 30%. In the most represented group, it decreased from 226 to 134. Additionally, the ratio of the mean size area to the highest (upper) area occupied by the clusters approached 1 (from 0.21 to 0.74) owing to the use of the additive, which indicated the suppression of the formation of larger clusters. All of the aforementioned effects helped in reducing the TSA value to the required level.

The selected samples were also tested using the spot test method (Figure 11, top row). The original sample of the marine fuel without the additive was rated least stable under category 5 according to ASTM D4740. A dark-glossy central area, which was much darker than the background, was observed in this sample.

The visualized spot test results were modified using software (Figure 11, bottom row) for the subsequent calculation of the

**Figure 7.** Marine fuels containing the additive (stable samples).

**Figure 8.** Comparison of marine fuels using microscopic analysis.
v-value. For this purpose, it was necessary to create red-green-blue (RGB) histograms (Figure 12).

The conversion into the RGB histograms (Figure 13) confirmed our subjective evaluation of the software-modified spot test results, in terms of changes in the representation of the blue color. The decrease in the TSA value was accompanied by a decrease in the blue color saturation in the modified spot test results. This effect was visible in the RGB histograms on decreasing the percentage maxima of the blue peak and shifting its position on the x-axis toward lower values (Figures 12 and 13). The x-axis represents the power of the color shade, with values ranging from 0 (the darkest shade of the color) to 255 (the lightest shade of the color).

The blue peak maxima moved from the color power value of 222−211. The percentage of blue tones decreased from ∼26 to ∼17%. Additionally, the distribution of the blue tones changed. Data obtained from the RGB histograms were used to calculate the v-value as an indicator of the colloidal stability of marine fuels. A v-value of 29 confirmed that the original sample was highly unstable. The sample with an additive content of 0.1 wt % was evaluated as unstable (v-value = 20), and the last sample
with an additive content of 2 wt % was characterized as stable ($\nu$-value = 12).

2.3. Final Product. The validated effective dosage of the additive was used for the laboratory preparation of the final marine fuel in large amounts (5 kg of each sample). The prepared marine fuels were analyzed in detail according to the requirements of the ISO 8217 standard (Table 2). The prepared fuels were stored for 60 days to verify the stability of the TSA parameters.

All of the required properties were successfully improved using the described additive, containing 30 wt % naphthalene. There was a slight increase in the density, viscosity, and micro carbon residue. There was an insignificant decrease in the flash point and content of the selected metals. The ash content and the total acid number (TAN) were at the same level. The most significant changes were observed in the TSA parameters.

Owing to the effects of the additive, both the prepared marine fuels (coded as B and C) met the requirements of the ISO standard. Moreover, the TSA value remained stable even after 60 days of storage.

3. DISCUSSION

We investigated the positive effects of an LCO as a refinery stream and a newly developed additive containing 30 wt % of naphthalene on the colloidal stability of marine fuels, especially the TSA parameter. The LCO was advantageous in that it was easily available as a lower-quality refinery intermediate, which shortened the life cycle of catalytic systems on hydrogenation units.28 Moreover, the LCO contained more sulfur components.29 These mainly included substituted DBTs derived from

Table 2. Detailed Characterization of the Laboratory-Prepared Marine Fuels Containing Effective Levels (2–3 wt %) of the Additive

| marine fuel | A    | B    | C    | ISO 8217 |
|-------------|------|------|------|----------|
| additive content (wt %) | 0    | 2    | 3    |           |
| naphthalene content (wt %) | 0    | 0.6  | 0.9  |           |
| density at 15 °C (kg·m⁻³) | 1004 | 1008 | 1010 | max. 1010 |
| K. viscosity at 50 °C (mm²·s⁻¹) | 645  | 660  | 690  | max. 700  |
| TSA (wt %) | undetectable | 0.08 | 0.05 | max. 0.10 |
| TSA (wt %) after storage | undetectable | 0.09 | 0.05 |           |
| MCRT (wt %) | 18.5 | 19.3 | 19.6 | max. 20   |
| flash point (°C) | 88   | 84   | 82   | min. 60   |
| Al + Si content (mg·kg⁻¹) | 7.95 | 7.91 | 7.90 | max. 60   |
| V content (mg·kg⁻¹) | 256  | 244  | 235  | max. 450  |
| Na content (mg·kg⁻¹) | 22.4 | 21.5 | 20.9 | max. 100  |
| ash content (wt %) | 0.08 | 0.07 | 0.07 | max. 0.15 |
| total acid number (mg KOH·g⁻¹) | 0.05 | 0.05 | 0.06 | max. 2.5  |

“Mentioned requirements correspond to the ISO-F-RMK category.

Figure 12. RGB histograms of the selected samples (R = red; G = green; B = blue).

Figure 13. Comparison of the blue histograms.
Table 3. Qualitative Parameters of Refinery Streams

| parameter | VR VBU | AGO VBU | LCO |
|-----------|--------|---------|------|
| density at 15 °C (kg·m⁻³) | 1071 | 845.8 | 988.2 |
| kinematic viscosity at (m²·s⁻¹) | 280 (150 °C) | 2.15 (40 °C) | 2.87 (40 °C) |
| monoaromatics (wt %) | N/A | 23.8 | 10.9 |
| diaromatics (wt %) | N/A | 8.4 | 72.4 |
| polyaromatics (wt %) | N/A | 0.6 | 9.0 |
| saturates (wt %) | 6.0 | N/A | N/A |
| aromatics (wt %) | 46.7 | N/A | N/A |
| resins (wt %) | 24.8 | N/A | N/A |
| asphaltenes (wt %) | 22.5 | N/A | N/A |
| alkanes (rel %) | N/A | 58.4 | 3.3 |
| cycloalkanes + alkenes (rel %) | N/A | 27.4 | 0.5 |
| diaromatics + benzothiophenes (rel %) | N/A | 0.6 | 87.1 |
| monoaromatics (rel %) | N/A | 13.6 | 5.9 |
| triaromatics + dibenzothiophenes (rel %) | N/A | 0.0 | 3.2 |

The relatively high sulfur content was the key limiting factor during the production of low- or ultra-low-sulfur marine fuels. However, the LCO, as a component of marine fuels, was still applicable to high-sulfur marine fuels or to low-sulfur marine fuels in a specific content, depending on the input matrix and other cutter stocks. However, a persistent technological problem resulted in a higher water content. Excessive foaming and overflow of the material from the expedition tank may occur during the production of marine fuels with the LCO. Therefore, it was necessary to ensure the sufficient removal of water from the LCO stream.

These bottlenecks were eliminated using a newly developed additive replacing the LCO. The additive contained 30 wt % unsubstituted naphthalene as the simplest diaromatic hydrocarbon. Technical issues associated with the high point of crystallization, poor solubility, and difficult applicability were addressed using a solution of naphthalene in tetralin. This concentrated the naphthalene from the initial 0.62 rel % (according to the GC × GC-TOFMS profile in Figure 3; retention time 1290 s) in the LCO stream to 30 wt % in the additive, which resulted in a reduction of the effective dose from 3 wt % for LCO to 1.5 wt % for the additive. This reduction in the effective dose indicated that unsubstituted naphthalene had a greater effect on improving the colloidal stability compared with other diaromatics, such as substituted naphthalenes, biphenyls, dibenzothiophenes, or fluorenes present in the LCO fraction. The effectiveness of the additive based on the substituted (alkyl) naphthalene has also been studied by other research teams in the past; however, it has been found that this additive (coded as VNII NP 200) loses its effect if the fuel contains a large number of unstable components. This information confirms our finding of a higher effect of unsubstituted naphthalene on the stability of marine fuels.

The use of the new additive was tested under laboratory conditions to verify the positive effect on the reduction of the TSA parameter. The TSA parameter is one of the key parameters often considered by marine fuel producers and users. A step change of this parameter was confirmed, owing to which the unsaleable material became compliant with stability requirements. The change in the TSA parameter was also accompanied by other indicators of colloidal stability. The set of adequate methods was used to confirm and describe the positive effects of the additive. Microscopic analysis was considered a suitable method for this purpose. In addition, as tools for stability comparison, the results of the spot test evaluation by RGB histograms, and consequently, the v-value parameters, were found to be in accordance with the existing literature.

The greatest bottleneck in the use of this new additive could be its relatively high price in relation to the price of produced marine fuel, which is often perceived by producers as rather a necessary waste. In this respect, however, the configuration and strategy of the individual refinery–petrochemical complexes as well as the ever-changing marine fuel market will play a major role.

4. CONCLUSIONS

The positive effects of the diaromatic hydrocarbon content on the colloidal stability of marine fuels were experimentally confirmed. The effects of LCO addition and a newly developed additive based on unsubstituted naphthalene were compared. The higher effect of unsubstituted naphthalene was confirmed using a set of prepared samples. The effective dose required to adjust the TSA parameter below the set limit of 0.1 wt % was reduced using this additive by 50% (1.5 wt %) in comparison with LCO (3 wt %). The additive had a positive effect on the TSA parameter, which is one of the most frequently required parameters by marine fuel producers and users. The additive converted the unsaleable fuel (TSA parameter could not be determined) into fuel that met the requirements set by the ISO 8217 standard. In this case, the TSA parameter was 0.08 wt % with using the additive in the concentration of 1.5 wt %. The positive effect of the additive on the colloidal stability of marine fuel was also verified and confirmed by a set of adequate analytical methods such as optical microscopy, spot test, or v-value parameter. The v-value parameter was reduced from the original value 29–12 using the additive, which indicates a very stable sample. The long-term (60 days) stability of prepared samples was verified as well. This study will be extended in the future to assess the possibility of using the additive for marine fuels containing biocomponents. The developed additive can be used for industrial applications in marine fuel production processes.

5. MATERIALS AND METHODS

A vacuum residue from the visbreaking unit (VR VBU) was used as the main component of the marine fuels. It was prepared on a laboratory scale. Atmospheric gas oil from the visbreaking unit (AGO VBU) served as a cutter stock. Light
Table 4. Analytical Methods

| analytical methods and parameters | standard | instrument |
|-----------------------------------|----------|------------|
| density                           | ASTM D4052 | DA 645 KYOTO ELECTRONICS |
| kinematic viscosity               | ASTM D445 | HVM 472 Herzog |
| aromatics content (HPLC)          | IP 391    | Agilent 1260 Infinity |
| total sediment accelerated (TSA)  | ISO 10307-2 | Setaclean Total Sediment Tester |
| saturates, aromatics, resins, and asphaltene (SARA) content | IP 469 | Iatroscan MK-6 |
| GC × GC-TOFMS simulated distillation (SimDist) | internal procedure | LECO Pegasus 4D GC × GC-TOFMS |
| microscopy analyses               | internal procedure | Leica DM2500 LED |
| spot test                         | ASTM D4740 | Memmert UF 30 Plus |
| v-value                           | Vráblík et al., 2018 | ImageJ software (NIH) |
| micro carbon residue testing (MCRT) | ISO 10370 | MCRT-160 MCRT TESTER PAC |
| flash point                       | ISO 2719  | ERAFLASH S10 |
| metal content                     | internal procedure | Agilent 725 ICP OES spectrometer |
| ash content                       | ISO 6245  | TGA 801SMC, LECO |
| total acid number (TAN)           | ASTM D664 | METTLER DL 25 |

“Test method is applicable to petroleum products and fractions with a final boiling point of 538 °C (AGO VBU and LCO).”

All of the analytical methods and parameters employed, including the equipment used, are listed in Table 4.

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**Author Contributions**

A.V. contributed to investigation, writing—original draft, methodology, software, data curation, validation, and resources; D.S. contributed to writing—original draft, software, visualization, and data analyses; K.D.J. contributed to data curation, visualization, and resources; J.M.H.H. contributed to writing—review and editing and supervision; and R.C. contributed to writing—review and editing and supervision. All authors have read and agreed to the published version of the manuscript.

**Notes**

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