

Effect of Ultrasonic Irradiation on On-board Fuel Analyzed Using Gas Chromatography/Mass Spectrometry

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GC/MS를 이용한 선박연료유에 대한 초음파조사 효과 분석

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Abstract : Since the enforcement of strict regulations on marine fuel oil sulfur content, demand for Low Sulfur Fuel Oil (LSFO) has been increasing. However, as LSFO properties vary greatly depending on the supply timing, region, and supplier, LSFOs can experience problems with sludge formation, blending compatibility, and stability once mixed into storage tanks. This study investigates using ultrasound cavitation effects to improve the quality of LSFOs in storage tanks. For marine gas oil (MGO), the results showed that the relative ratio of high molecular weight compounds to those of low molecular weight decreased after ultrasonic irradiation, due to cavitation-induced cracking of chemical bonds. For marine diesel oil (MDO) and blended oil, a small increase in the relative abundance of low weight molecular compounds was observed after treatment. However, no correlation between time and relative abundance was observed.

Key Words : Low Sulfur Fuel Oil (LSFO), Blended fuel, Ultrasonic energy, Cavitation, Gas Chromatography/Mass Spectrometry (GC/MS)

요 약 : 최근 선박용 연료유에 대한 황 함유량 규제를 준수하기 위해 저유황유의 수요가 증가하고 있다. 그러나 저유황유를 공급하는 시기, 지역, 회사 별로 그 품질이 상이함에 따라 선내 연료유 저장탱크에서는 과도한 슬러지가 발생하는 등 혼합 안정성에 대한 문제가 제기되고 있다. 따라서 본 연구는 초음파의 캐비테이션 현상을 이용하여 저유황유의 품질 향상을 하고자 하였다. 선내 저장 탱크에서 이종의 연료유가 혼합되는 상황을 모사하기 위해 두 가지 종류의 저유황유(황 함유량 0.5% 이하 MGO, MDO)를 혼합하여 시료유로 사용하였다. 원료유와 50 wt.% 씩 혼합한 시료유를 120분 동안 초음파 처리하였으며, 40분 주기로 채취된 샘플은 GC/MS 분석을 수행하여 초음파 조사 시간에 따른 시료유의 조성 변화를 분석하였다. 연구결과, 초음파의 캐비테이션 효과로 인하여 화학결합이 깨지면서 MGO 내 존재하는 고분자량 화합물의 감소와 저분자량의 화합물 증가가 관찰되었다. MDO와 혼합유의 경우, 초음파 조사 후 저분자 화합물에 대한 상대 존재비의 부분적 증가가 관찰되었지만 시간과 상대 존재비 사이의 상관관계는 관찰되지 않았다.

핵심용어 : 초저유황유, 혼합연료유, 초음파 에너지, 공동현상, 가스크로마토그래피/질량분석기

1. Introduction

According to international regulations, all ships on international voyages must use only fuel with a sulfur content of 0.5 % or less, and only use fuel oil with a sulfur content of less than 0.1 %, which is a stricter standard, in emission control areas (ECA) (IMO, 2016). Shipping companies in each country are using methods such

as the use of low sulfur fuel oil (LSFO), installation of desulfurization facilities such as scrubbers in ships, and the introduction of liquefied natural gas (LNG) fueled ship in order to respond to international regulations. The shipping companies prefer to use low sulfur fuel oil because desulfurization facilities such as scrubbers incur additional cost and it is not easy to be applied to other ships except target ship in the case of LNG fueled ship (Lee and Ryu, 2019). As the consumption of LSFO is expected to increase rapidly, large refineries and crude oil suppliers around the

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world are continuously expanding their LSFO production facilities.

Hydro-desulfurization (HDS) technology, which is the most widely used technology for low sulfur oil production, can effectively remove low molecular weight sulfur compounds. However, the deep-desulfurization process, which removes high molecular sulfur compounds, requires a higher temperature and pressure than conventional processes and has been a problem because of continuous hydrogen consumption (Javadli and Klerk, 2012). Recently, oxidative desulfurization (ODS) technology, which is a method of extracting sulfur by oxidation with sulfur dioxide (SO₂), has been used instead of the deep-sulfurization process (Filippis and Scarsella, 2003). Although these methods can effectively remove sulfur in fuel oil, they can be applied only to some large refineries due to facility cost. Most other fuel suppliers are supplying blended LSFO by mixing low-sulfur oil and high-sulfur oil.

Physically mixed LSFO has caused quality problems and it has been reported that more than 55 % of shipping companies experienced fuel quality problem (BIMCO et al., 2020). Inadequate mixing of different fuel oils generates excessive sludge and reduces mixing and storage stability (Lee and Kim, 2019). Due to the characteristics of the ship operation where the bunkering location is not constant, low sulfur oil of different quality can be mixed with the oil remaining in the onboard storage tank. Blending is regarded as simple process in a physical point of view, but the properties of fuel oil vary greatly depending on the production time of fuel oil and the method of refining, so several factors must be considered when oil mixing.

Polymer compounds such as sludge produced by improper mixing cause blockage of pumps and filters in the fuel system, and increase air pollution due to incomplete combustion and inappropriate fuel oil supply to the fuel injection system. As a physical treatment to remove polymer compounds from fuel oil, adoption of purifier brings a solution. Due to problems such as the use of low sulfur oil manufactured by a heterogeneous fuel oil mixing and the generation of sludge induced by the irregular mixing of residual oil in the ship, the ship might be asked to install.

Cavitation is a phenomena formed as a pressure change in fluids due to velocity change. When a liquid moves at a high speed, the pressure of the liquid is lowered below the vapor pressure and

vapor bubbles are generated in the liquid (Brennen, 1995). At the point where the cavity collapses, high temperature (5000 °C) and high pressure (1000 atm) is instantaneously formed (Jafari et al., 2018). This promotes chemical reaction by breaking or bonding the chemical bonds of molecules and generating free radicals in the process (Bolla et al., 2012). In addition, it is known that a micro emulsion is generated between the interfaces of heterogeneous fluids. Recently, researches using ultrasonic energy for fuel oil have been continuously published such as UAOD (ultrasonic assisted oxidative desulfurization) research that increases the efficiency of desulfurization by accelerating the oxidation reaction of a fluid by using the ultrasonic cavitation effects (Margeta et al., 2016; Zhou et al., 2020).

This study was conducted to improve the storage and mixing stability of LSFO on ships by decomposing high molecular weight compounds into low molecular weight compounds using the cavitation effects of ultrasonic energy. For this purpose, the change of chemical species for MGO (marine gas oil), MDO (marine diesel oil), and the mixed oil was observed according to the ultrasonic irradiation time. MGO is a pure distillate oil with low turbidity and is commonly referred to as marine diesel. MDO is manufactured by mixing light oil (MGO) and heavy oil (Bunker C oil), and the mixing ratio of MGO and heavy oil is about 90:10. Various of fuels applicable to ships are bunker C, MDO, MGO, etc., but bunker C is not a low sulfur oil so it was excluded from this experiment. Fuels were pretreated and analyzed using gas chromatography mass spectrometry (GC/MS).

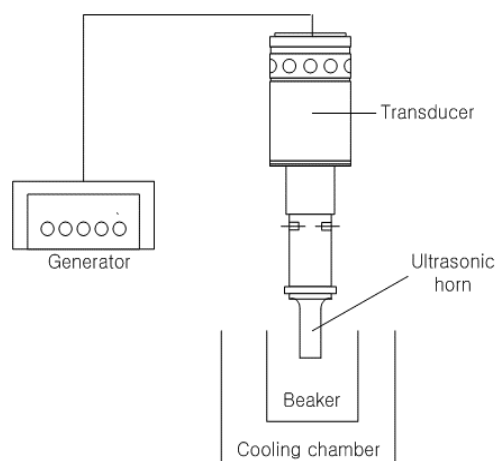


Fig. 1. The schematic of ultrasonic experiment equipment.

2. Method and experiment

2.1 Ultrasonic equipment

A schematic diagram of the ultrasonic device is presented in Fig. 1. The device is composed of an oscillator, a transducer, and a horn type vibrator. The vibrator is directly exposed to sample oil to effectively irradiate the ultrasonic. The maximum power of the ultrasonic equipment is 600 W and the frequency is 19.8 kHz. The beaker was inside of the cooling chamber to keep sample fuel temperature constantly (around 4 °C).

2.2 Experiments

MGO, MDO and a blend of two fuels were used. Two types of low-sulfur oil were mixed and used as sample to simulate the mixing of different fuel oils in the onboard storage tank. The blended oil was prepared by mixing MGO and MDO at a mixing ratio of 50 wt.%. For homogeneous mixing of the sample oil, it was stirred for about 30 min at 1000 rpm using a magnetic stirrer. The ultrasonic horn was directly exposed in 500 ml of sample oil, and the temperature of the sample oil was kept constant at a level of 50 to 60 °C. The experiment was carried out continuously for 120 min, and samples were taken every 40 min for analysis. The amount of each sample was 2 ml, and the effect of reducing the total amount of sample oil due to sampling was neglected.

2.3 Analytical methods using GC/MS

Analysis was performed at the analysis center in the Korea Advanced Institute of Science and Technology (KAIST) to compare the chemical species of the sample oil. The fuel oil was extracted with dichloromethane (DCM, above 99.8 %), and the dilution ratio was adjusted to obtain a significant measured value. ISQ-QD300 as GC/MS by Thermo fisher was used, and it consist of an automatic injection device, a split/non-split injector, an oven, and a single quadrupole mass spectrometer.

Table 1 shows the GC/MS analysis conditions. TG-5MS column (Thermo scientific) which is non-polar and stable for heating was used and it is composed of 5 % phenylmethyl polysiloxane. Helium (He) gas was used as the mobile phase gas, and the flow rate was 1 ml/min. After maintaing the initial temperature of the column at 40 °C for 5 min, the temperature was raised to 300 °C at a rate of 10 °C per min, and the final temperature was allowed to stay for 10 min. Electron ionization (EI) was used as the

ionization method in the mass spectrometer, and the temperature of the ion source was 280 °C.

3. Results and discussion

3.1 Characteristic of fuel oil before ultrasonic irradiation

The GC/MS analysis result of MGO, MDO, and mixed oil before ultrasonic irradiation were presented in Fig. 2. The x-axis is time (run time), and the y-axis is the signal of each peak converted to an area fraction (Kim et al., 2019).

Table 1. GC/MS operating conditions

GC Conditions	
Column	TraceGoldTG-5MS (10 m × 0.1 mm × 0.1 μm)
Inlet Temperature	300 °C
Inlet Mode	Split
Column flow	1 mL/min (constant flow)
Oven Initial Temperature	40 °C for 5 min
Ramp #1 rate	10 °C/min
Ramp #1 final temperature	300 °C
Ramp #1 hold time	15 min
Run time	43 min
MS Conditions	
Ion Source temperature	280 °C
Data acquisition delay	3 min
Mass range	5 – 1050 m/z

The relative ratio was to compare the composition ratio of chemical species for each oil type, and was calculated by calculating the ratio of the area sum of all chemical species to the area of the target chemical species.

The ratio of n-alkane (C_nH_{2n+2}), a chain saturated hydrocarbon, was calculated for each fuel oil and shown in Fig.3. Normal alkanes are observed in the range of C_7 (heptane) to C_{27} (heptacosane), and it can be seen that the ratio of high molecular compounds exceeding C_{19} is little bit high in MDO. It is due to residual oil (Bunker C) about 10 % in MDO. Bunker C is a dark brown viscous oil obtained after distilling gasoline, kerosene, light oil, etc. from crude oil. In MGO, relatively low molecular weight alkanes (C_{11} ~ C_{19}) were analyzed more than MDO, and about 16 % more higher peak area than the MDO analyzed (Fig. 3).

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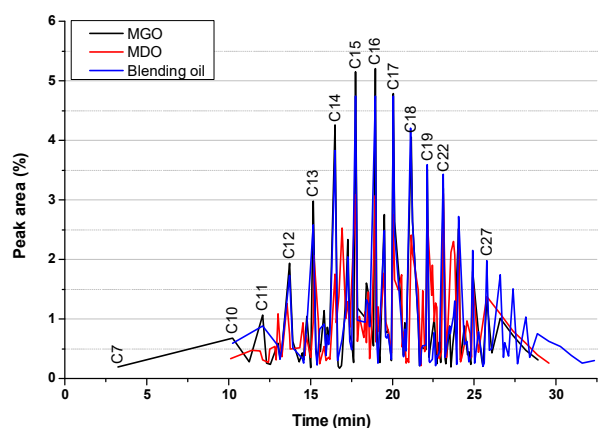


Fig. 2. GC/MS result for MGO, MDO and blending oil before ultrasonic irradiation.

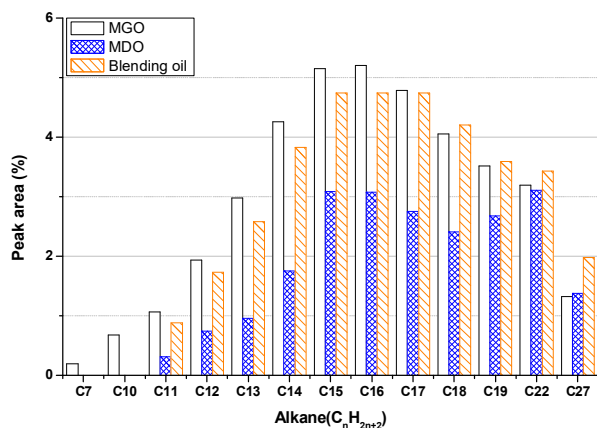


Fig. 3. The relative ratio of alkane in MGO, MDO and blending oil before ultrasonic irradiation.

3.2 Characteristic of fuel oil after ultrasonic irradiation

3.2.1 MGO (marine gas oil)

Fig. 4 and Table 2 shows results of GC/MS analysis for MGO oil according to the ultrasonic irradiation time, and it is expressed as a bar graph by dividing into one group including each alkane and all chemical species included between the alkanes. For example, $\sim C_{12}$ is the sum of all hydrocarbons observed after C_{11} and the peak area of $C_{12}H_{26}$ divided by the total area of the peak. (For description, $\sim C_n$ is expressed as $C_{n\text{-sum}}$)

As shown in the figure, as the ultrasonic irradiation time increased, high molecular weight compounds exceeding $C_{16\text{-sum}}$ decreased, and low molecular weight compounds less than $C_{14\text{-sum}}$ increased. The relative proportion of $C_{10\text{-sum}}$ before ultrasonic

irradiation was 0.68 %, but after ultrasonic irradiation, it increased to 0.71, 0.80, and 2.03 % (time: 40 min, 80 min, and 120 min). In the case of $C_{17\text{-sum}}$, the largest decrease was observed, and 12.16 % for $C_{17\text{-sum}}$ before ultrasonic irradiation was observed to be decreased to 12.00 %, 11.08 %, and 10.85 % (time: 40 minutes, 80 minutes and 120 min). The relative abundance of $C_{7\text{-sum}} \sim C_{14\text{-sum}}$ increased within the range of 0.34 % \sim 1.34 %, and the relative abundance of $C_{15\text{-sum}} \sim C_{27\text{-sum}}$ decreased within the range of 0.29 % \sim 1.31 %. The total chemical species below $C_{14\text{-sum}}$ increased from 22.59 % to 23.34 %, 25.0 %, and 27.11 %, and those above $C_{15\text{-sum}}$ decreased from 74.46 % to 73.70 %, 71.61 %, and 69.40 %.

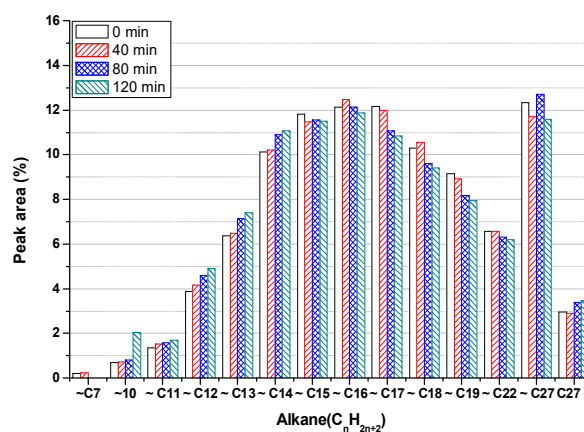


Fig. 4. The relative ratio of the hydrocarbon($C_{n\text{-sum}}$) in MGO with ultrasonic irradiation.

High molecular compounds decomposed by ultrasonic energy generate to low molecular species (ex: $C_{n\text{-sum}} \rightarrow C_{n-i\text{-sum}} + C_{n-j\text{-sum}} + \text{etc.}$, here $i, j > 0$), at the same time, target chemical species (ex: $C_{n-i\text{-sum}}$) are simultaneously decomposed ($C_{n-i\text{-sum}} \rightarrow C_{n-i-k\text{-sum}} + C_{n-i-l\text{-sum}} + \text{etc.}$, here $k, l > 0$) by ultrasonic energy. Although it was not possible to confirm a certain ratio relationship between the increase and decrease of the relative abundance of chemical species, it was found that the decomposition rate of the high molecular compound was slightly higher than that of the low molecular compound.

Due to the cavitation effects by ultrasonic, the high molecular compound was cracked into small molecules so decrease in the relative abundance of the high molecular compound was observed. When aliphatic hydrocarbons are sonicated, decomposition of chemical species occurs and decomposed into hydrocarbons with a smaller structure.

It has been reported that decane ($C_{10}H_{22}$) is decomposed to hydrogen, methane (CH_4), acetylene (C_2H_2), ethylene (C_2H_4) and propylene (C_3H_6) during ultrasonic irradiation (Suslick et al., 1983). Aromatic and naphthenic hydrocarbons with large binding energy are also decomposed through ultrasonic, and benzene (C_6H_6) and toluene (C_7H_8) aromatic hydrocarbons are decomposed into hydrocarbons such as acetylene (C_2H_2) (Cataldo, 2000). At a frequency of 40 kHz, part of asphaltene was decomposed, which is known to be caused by radicals ($\cdot OH, \cdot H$) generated during ultrasonic irradiation (Sadeghi et al., 1990). At this time, hydrophobic and volatile compounds are mainly decomposed by radical and thermal decomposition reactions occurring in the gas-liquid interface region and inside the bubble, and hydrophilic and non-volatile compounds are decomposed by the tendency to accumulate in the liquid phase (Psilakis et al., 2004).

Table 2. Relative abundance of $C_{n\text{-sum}}$ in MGO with ultrasonic irradiation

Time (min)	Hydrocarbon relative abundance (%)			
	0	40	80	120
$C_{7\text{-sum}}$	0.19	0.23	0	0
$C_{8\text{-sum}}$	0.68	0.71	0.80	2.03
$C_{9\text{-sum}}$	1.35	1.52	1.57	1.69
$C_{12\text{-sum}}$	3.87	4.16	4.60	4.90
$C_{13\text{-sum}}$	6.37	6.50	7.14	7.41
$C_{14\text{-sum}}$	10.13	10.22	10.90	11.08
$C_{15\text{-sum}}$	11.81	11.48	11.57	11.52
$C_{16\text{-sum}}$	12.14	12.47	12.15	11.88
$C_{17\text{-sum}}$	12.16	12.00	11.08	10.85
$C_{18\text{-sum}}$	10.31	10.55	9.61	9.42
$C_{19\text{-sum}}$	9.14	8.92	8.19	7.94
$C_{22\text{-sum}}$	6.57	6.56	6.30	6.19
$C_{27\text{-sum}}$	12.33	11.72	12.71	11.6

Fig. 5 and Table 3 shows only the proportion of alkanes in MGO, and all of the chemical species above C_{14} except for C_{27} shows decreasing relative abundance according to the ultrasonic irradiation time. After 40 min, C_{15} , C_{16} , C_{17} , C_{18} , C_{19} , and C_{22} alkanes decreased by 0.22 %, 0.23 %, 0.29 %, 0.11 %, 0.17 %, 0.07 %, respectively, compared to previous ultrasonic. After irradiation, it decreased by the proportions of 0.39 %, 0.82 %, 1.14 %, 1.11 %, 0.87 %, 0.80 %, 0.57 % (C_{14} , C_{15} , C_{16} , C_{17} , C_{18} , C_{19} and C_{22}), respectively. In contrast, it can be seen that alkanes

below C_{13} increased from 0.06 % to 0.3 %. The reduction of the high molecular compound becomes bigger as the ultrasonic irradiation time increases, and it is estimated that the chemical bond cracking occurs due to the cavitation effects by the ultrasonic.

Although the rate of decomposition is different depending on the compound, it can be seen that the reduction rate of C_{16} to C_{18} is the greatest between 40 and 80 min based on the ultrasonic irradiation time. When the ultrasonic irradiation time kept more than 80 min, low molecular compounds less than C_{10} were not detected, and high molecular weight compounds exceeding C_{27} were rather increased. Because free radicals are generated when a high molecular compound is decomposed into a low molecular compound. If the cavitation effects continues for a certain period of time or more, the rate at which the already generated radicals are recombined to the compound increases rather than the rate at which the compound is decomposed to form other compounds (Najafi and Amani, 2011).

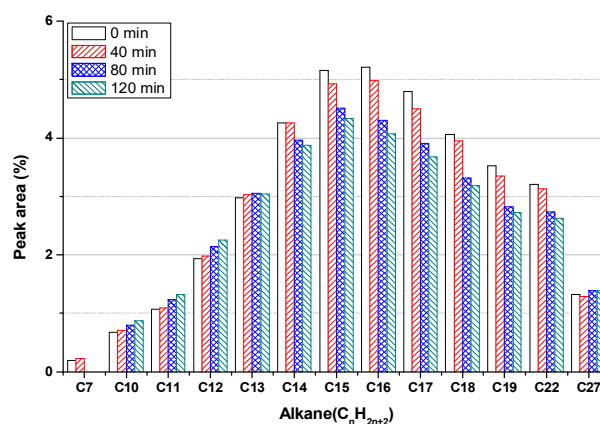


Fig. 5. The relative ratio of alkane in MGO with ultrasonic irradiation.

Changes in the species ratio of alkanes are clearly observed compared to changes in various compounds, which are cycloalkanes, multiple-bonded hydrocarbons, and aromatic hydrocarbons in fuel oils. The alkane species having less binding energy than other hydrocarbon and it makes more easy decomposition than others by ultrasonic energy. About 75 % of aliphatic hydrocarbons and 25 % of aromatic hydrocarbons exist in oil, and the alkane compound has the largest proportion (Handbook of air pollution from internal combustion engines, 1998; Huth and Helios, 2013).

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Comparing the bonding energy between carbons, the C-C bond is 331 kJ/mol, C=C is 614 kJ/mol, the hybrid structure is 518 kJ/mol, and the bonding energy between C≡C is 839 kJ/mol. Since the binding energy of an alkane is smaller than that of multiple bonds and aromatic hydrocarbons, it is more easily decomposed even with a small energy.

3.2.2 MDO & Blending oil

Fig. 6 shows results of GC/MS analysis for MDO according to the ultrasonic irradiation time, and it is expressed as a bar graph by dividing into one group including each alkane and all chemical species included between the alkanes. In contrast to MGO, no significant change in the composition of the compounds present in MDO was observed. A partial increase (0.07 to 0.33 %) of the relative abundance for low molecular compounds was observed after ultrasonic irradiation, but no correlation between time and relative abundance was observed.

Table 3. Relative abundance of C_n in MGO with ultrasonic irradiation

Time(min)	Hydrocarbon relative abundance (%)			
	0	40	80	120
C ₇	0.19	0.23	0.00	0.00
C ₁₀	0.68	0.71	0.80	0.87
C ₁₁	1.07	1.09	1.23	1.32
C ₁₂	1.94	1.98	2.15	2.25
C ₁₃	2.98	3.03	3.05	3.04
C ₁₄	4.26	4.26	3.96	3.87
C ₁₅	5.15	4.93	4.51	4.33
C ₁₆	5.21	4.98	4.30	4.07
C ₁₇	4.79	4.50	3.91	3.68
C ₁₈	4.06	3.95	3.31	3.19
C ₁₉	3.52	3.35	2.82	2.72
C ₂₂	3.20	3.13	2.74	2.63
C ₂₇	1.32	1.29	1.39	1.39

The correlation between the irradiation time and the relative abundance of the compound was not clearly observed as the decomposition and binding of the compound were repeated as the ultrasonic irradiation continued.

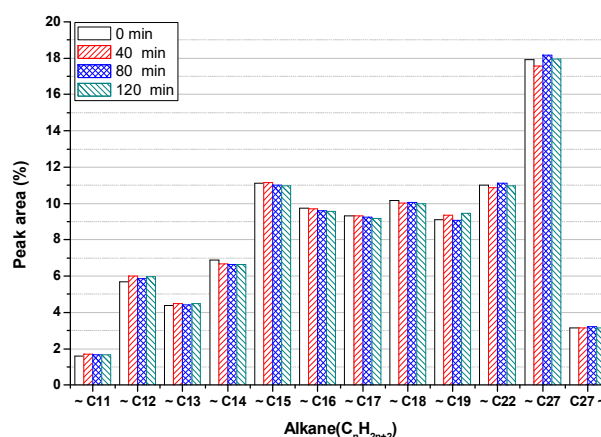


Fig. 6. The relative ratio of the hydrocarbon(C_{n sum}) in MDO on ultrasonic irradiation.

Fig. 7 shows results of GC/MS analysis for Blending oil according to the ultrasonic irradiation time. Similar to MDO, a change was not clearly observed according to the ultrasonic irradiation time.

Decomposition of high molecular compositions are governed by the frequency and the power of ultrasonic. It is known that the higher frequency and power of ultrasonic brings the better dispersion and decomposition molecular characteristics. PAH (polycyclic aromatic hydrocarbons) compounds are almost decomposed in about 60 to 120 min under irradiation conditions of 80 kHz, 150 W ultrasonic waves (Psilakis et al., 2004), and some asphaltenes in heavy oil were decomposed under irradiation conditions of 40 kHz (Sadeghi et al., 1990). The frequency of the ultrasonic equipment used in this experiment was 19.8 kHz, which may not be sufficient to completely decompose various polymeric compounds. Since it is effective for chemical species with small binding energy, it has a limited effect to the compounds which have high binding energy. In addition, since radicals generated by cavitation makes compound recombination (Najafi and Amani, 2011) and induce decomposition of the material in other parts at the same time, the ratio change of chemical species may not be observed to a noticeable degree.

Since the optimal irradiation time for compound decomposition depends on the characteristics of the sample oil (viscosity, surface tension, etc.), it was difficult to clearly define the effect of ultrasonic irradiation time on MDO and blending oil in the sampling cycle of this experiment.

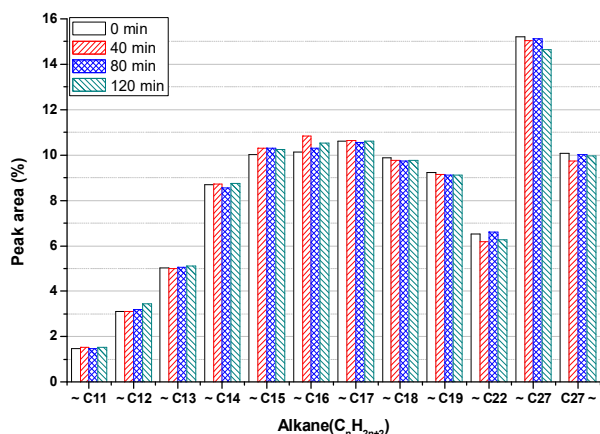


Fig. 7. The relative ratio of the alkanes($C_{n\text{-sum}}$) in blending oil on ultrasonic irradiation.

4. Conclusion

In this study, GC/MS analysis was performed on the relative abundance changes of compounds in MGO, MDO, and blending oil (MGO 50 wt.%, MDO 50 wt.%) according to the ultrasonic irradiation time, and the following results were obtained.

As the ultrasonic irradiation time increased, high molecular weight compounds exceeding $C_{16\text{-sum}}$ decreased, and low molecular weight compounds less than $C_{14\text{-sum}}$ increased. Changes in the species ratio of alkanes are clearly observed compared to changes in various compounds, which are cycloalkanes, multiple-bonded hydrocarbons, and aromatic hydrocarbons in fuel oils. The alkane species having a small binding energy is more easily decomposed by ultrasonic energy compared to other hydrocarbons.

In contrast to MGO, no significant change in the composition of the compounds present in MDO and blended oil was observed. A partial increase of the relative abundance for low molecular compounds was observed after ultrasonic irradiation. The correlation between the irradiation time and the relative abundance of the compound could not be clearly defined because the decomposition and new binding of compound repeatedly occurs during the ultrasonic irradiation.

It is known that the physicochemical change of a compound by ultrasonic irradiation is affected by ultrasonic power, reaction time, and temperature. It is reported that the most influencing parameter is the power of ultrasonic energy, followed by reaction time and temperature (Shi et al., 2017). The frequency of the ultrasonic equipment used in this experiment is 19.8 kHz, which may not be sufficient to completely decompose various polymeric

compounds including polyaromatic hydrocarbons. Since radicals generated by cavitation recombine with the compound (Najafi and Amani, 2011) and at the same time induce decomposition of the material in other parts, the portion of chemical species changes may not be observed to a noticeable degree.

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