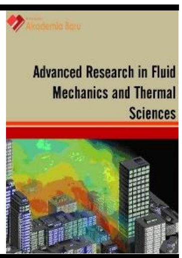
Penerbit Akademia Baru

Journal of Advanced Research in Fluid Mechanics and Thermal Sciences

Journal homepage: www.akademiabaru.com/arfmts.html
ISSN: 2289-7879



Characterization of Vehicle Smart Fluid using Gas Chromatography-Mass Spectrometry (GCMS)

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ARTICLE INFO

Article history:
Received 22 January 2019
Received in revised form 20 February 2019
Accepted 2 March 2019
Available online 18 March 2019

ABSTRACT

The great properties of magnetorheological (MR) fluid have amplified the request to create more type of MR fluid. Despite numerous studies suggesting many formulations in this area, their role in the vehicle industry is still far from ready for any practical applications. In this study, individual components of the carrier fluid in an original equipment manufacturer (OEM) smart fluid preferably acknowledged as MR fluid that contributes to the development of stress has been investigated. The carrier fluid components were identified through gas chromatography (GC) techniques coupled with mass spectrometry (MS) analysis over a range of retention time and computer matching with the National Institute of Standards and Technology (NIST) library. The data is statistically analysed to predict the potential material use as a carrier fluid in MR fluid. It was found that a total of seven components with a noble match and high probability were successfully identified. The MR fluid exhibited oily properties were related to the presence of alkane hydrocarbons components, of which Heptadecane (0.98%), Nonadecane (0.61%), Tetratetracontane (1.72%) and Eicosane (7.94%) respectively. The results also revealed that the number of additives present in the mixture was more than one with a higher concentration percentage. The additives have a major influence on the overall magnetorheological effects of the MR fluid.

Keywords:
smart fluid, magnetorheological fluid, gas chromatography, mass spectrometry, carrier fluid

1. Introduction

The vehicle suspension system is designed with compromised performance between ride comfort and vehicle handling. Excellent performance of the suspension system can be attained by implementing a controllable damper where its damping properties can be remotely adjusted. Smart fluid with its rheological property varied by the exterior stimulus is an ideally convenient working fluid for this group of the damper. Research on smart fluid preferably magnetorheological (MR) fluid has a long history. MR fluid was found at the end of the 1940s and early 1950s. However, interest in MR fluids languished until the early 1990s when advancements in MR material and the advent of

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readily available digital control electronics fostered renewed interest. Research in magneto rheology has gone forward in three main areas which are characterizing the mechanism that causes the MR effect, applications of MR materials, new structures of MR fluid and its influencing factors. Advancement in these areas is necessary for the development of mechanical systems that will optimize MR materials. MR fluid is an old-new comer of damping material that has surpassed its counterpart electrorheological (ER) fluid in the wide range of application due to its ability to harvest ten times stronger shear strength. The application of MR fluids decrees its formulation which typically a suspension of permeable particles that can be magnetised in a non-magnetic carrier fluid. The suspended magnetic particles appear to align or cluster creating a “necklace” look like structure when a magnetic field is induced. The fluids behave in a more viscous manner as the “necklace” needs to be “broken” in order to flow. The minimum force needed to break the “necklace” is known as yield stress. In the absence of magnetic field, MR fluid exhibits ideal Newtonian behaviour with an acceptable viscosity in the function of carrier fluid properties, particles size and volume fraction. In an instant, these induced rheological properties are completely reversible [1–3].

Oils are by far the most commonly used carrier fluids for MR fluid as shown in Table 1. Low viscosity advantages of oils make them the dominant candidate in the selection of MR fluid carrier fluid. Carrier fluids are often referred to as liquids previously developed to suspend the particles. The compromise between low viscosity and low vapour pressure provides ideal rheological properties and temperature stability. Low viscosity is needed to maximize the MR effect. In turn of events, it prompts to the instability of the fluid whereas high viscosity raises the concern of undesirable viscosity in absence of magnetic field. An additional key aspect of carrier fluid is to have a low vapour pressure as it is an increasingly important property for MR fluid wide range of temperature application.

Table 1
Commonly used carrier fluid type and its viscosity [4]

Carrier fluid type	Viscosity at 40°C ($Pa.s$)	Flash point (°C)
Mineral oil	0.0280	171-185
Synthetic oil	0.1068	230
Silicone oil	0.1100	More than 300

The research interest in the selection of carrier fluid among the scientist in this field increase because of its significant effect on MR fluid rheological characteristic. A large and growing body of literature has investigated polymer as a new type of carrier fluid for MR fluid. In this context, the first systematic study of polysiloxane was undertaken by Xie *et al.,* in which they proposed polysiloxane based elasticity cement as the carrier fluid. Polysiloxane, more precisely called polymerized siloxane consist of an inorganic silicon-oxygen backbone chain with organic side groups attached to the silicon atoms. These silicon atoms are tetravalent. They reported the mixture exhibit strong MR effect exceeding the limit of the rheometer they used [5]. In a follow-up study, Xie *et al.,* found that the suspension has great stability against sedimentation up to more than three months [6]. In the same year, Vasiliev *et al.,* published a paper in which they developed a hyperbranched polycarbosilane based MR fluid. Despite the fact, hyperbranched polycarbosilane has a higher molecular mass, they observed that the composition lower than 72% mass of magnetic filler demonstrate Newtonian fluid behaviour [7].

A considerable amount of literature has been published on using a new generation of magnetic suspension known as magnetorheological (MR) gel with higher viscosity properties. MR gel has been identified as a major contributing factor for the decline of the sedimentation rate of magnetic particles. Previous research has indicated polymeric gel deposition on the iron particles surface have a positive impact on the stability of the system [8]. However, there is a conflict of interest of viscosity during off-state condition. In these regards, a recent study of the magnetorheological gel by Zhang *et al.,* involved polytetrafluoroethylene (PTFE) micro powders to fabricate PTFE-oil organogel. They observed there was a significant viscosity change with an increase of PTFE contents. They also found that the critical PTFE volume fraction was 4.7 vol %. Unfortunately, the magnetization saturation of the carbonyl iron decreased and hinders the induced chain [9]. On the grounds of using aqueous suspension, Peer *et al.,* examined the stability of polyethylene oxide under microwave-assisted radiation. Their results showed the mixture stability is strengthened because the presence of polyethylene oxide promotes the formation of nanofibrous webs [10].

Interest in preparing MR fluids based on ionic liquids (ILs) increasing because of its alterable properties. ILs which mainly made from ions can modify its viscosity, solubility, electrical conductivity and melting point by considering the composition of its ions. Different from conventional carrier fluids, ILs have high stability and sustainable due to its state of liquids and negligible vapour pressure and flammability [11]. In this esteem, Guerrero-Sanchez *et al.,* became the first researchers to produce an ionic liquid-based MR fluid. Their investigation showed that there is no undesirable change in the MR effect. However, the rate of sedimentation is greatly reduced without any addition of additives [12]. Then Rodriguez-Arco *et al.,* did a survey on utilizing ILs for used in MR fluids. They provided a broader perspective stating that there still a lot of question on the validity employing ILs in MR fluid. ILs compatible dispersed phase and its concentration, relationship analysis of particle surface and constituent ions have not been studied thoroughly yet [13]**.**

In spite of the fact that numerous studies of carrier fluid involve monitoring its effect on the enhancement of yield stress and these fluids stability has been conducted, it is sensible to state that there is no ambiguous evidence on investigated materials and formulation found fulfilling the expectations for vehicle industrial applications as well as directly compete with LORD original equipment manufacturer (OEM) MR fluid. Therefore, further studies are needed in the context of identifying the essential carrier fluid component. With the rapid development of MR fluids, including LORD MR fluid commercial success in a luxury vehicle and suited to a wide variety of application, analysis of their essential carrier fluid component is important. An analytical technique known as gas chromatography-mass spectrometry (GCMS) that continue to enjoy important application in the food industry and medicine is one of the advanced separations and identification method today. The hybrid of separation capabilities of GC for volatile components in a sample and detection and identification attributes of MS on the basis of their mass is proven increased efficiency of sample analyses. GCMS is used almost exclusively for the qualitative analysis of the volatiles. With reference to Table 1, it is known that carrier fluid commonly used is volatile. The aim of this work was to investigate the components of the carrier fluid obtained from original equipment manufacturer MR fluid. To the best of our knowledge, no previous studies concerning their actual components using GCMS technique have been published.

2. Experimental

2.1 The MR Fluid

LORD MRF-122EG fluid was selected for the analysis. LORD MRF-122EG fluid is a hydrocarbon-based magnetorheological (MR) fluid formulated for general use in controllable, energy-dissipating applications such as damper and brakes.

2.2 Extraction of the Essential Carrier Fluid from MR Fluid

The MR fluid was subjected to a separation process using a centrifuge for 10 minutes and 1800 rpm at 24°C. In this process, the centrifugal force is used to promote accelerated settling of the magnetic particles in the MR fluid. The magnetic particles which are often seven to eight times dense than carrier fluid migrate away from the axis while the carrier fluid migrates towards the axis. The pellets (the magnetic particles) gather in the bottom of the tube while the supernatant (the carrier fluid) then discarded using a pipette. Two samples of 5ml were obtained.

The concentration of the carrier fluid extracted was decreased by adding hexane as an additional solvent. The dilution process can be shown in the following mathematical equation.

$$M_1V_1 = M_2V_2 \quad (1)$$

where, M_1 denotes the concentration of the original carrier fluid solution and V_1 denotes its original volume while M_2 represents the diluted solution concentration and V_2 represents its final volume. Since the original concentration of the carrier fluid is unknown, the dilution factor was used and the sample of the diluted carrier fluid was denoted as 10X and 100X.

The samples were further subjected to a filtration process. The samples were forced to pass through the pores of the filter to ensure the impurities completely remove from the samples. The samples were submitted to gas chromatographic analysis.

2.3 Gas Chromatography-Mass Spectrometry (GCMS) Analysis

(Figure 1) Gas chromatography was carried out on an Agilent 5975C TAD Series GC/MSD system equipped with an HP-5 (5% phenyl 95% dimethylpolysiloxane) fused silica (Agilent 19091S-433) capillary column ($30m \times 0.25\,mm$; film thickness $0.25\,\mu m$), the mass spectrometry detector temperature was set to 280°C. The sample ($1\mu L$, diluted 1:100 with hexane) was injected in a split mode ratio 5:1. The initial oven temperature was set to 70°C for a minute and then up to 185°C at $5\,°C/min$ and held again for a minute. It was further increased to 300°C at a same ramping rate as before. The carrier gas was helium at a constant flow rate of $3\,mL/min$.

Fig. 1. Agilent 5975C TAD Series GC/MSD system

2.4 Identification of Components

The components of the carrier fluid were identified by their retention time, retention indices relative to n-alkanes and computer matching with the National Institute of Standards and Technology (NIST) library. The concentration of the identified compounds was computed from the GC peak area. Relative percentages of the components were calculated based on the GC peak areas without a correction factor.

3. Results and Discussion

As shown in the Table 2 and illustrated in Figure 2, seven components were identified in the carrier fluid of the MR fluid. The major components were Bis(2-ethylhexyl) ester (50.25%), 2-propyl tetradecyl ester (37.42%) and eicosane (7.94%). In addition, MR fluid comprises five alkane hydrocarbons (11.25%) and three acids (88.75%); one mineral acid (37.42%) and two organic acids (51.33%) as portrayed in Figure 3. This finding is also tabulated systematically in Table 3. Therefore, the majority of the components found in the MR fluid carrier fluid were alkane hydrocarbons.

Table 2
Components of the MR Fluid (LORD MRF-122EG) obtained by GCMS on HP-5 column

No	Retention Time	Name	Formula	Probability (%)	Concentration (%)
1	6.438	Heptadecane	$C_{17}H_{36}$	78	0.98
2	15.345	Butyl propyl ester	$C_8H_{16}O_3$	90	1.08
3	25.623	Nonadecane	$C_{19}H_{40}$	95	0.61
4	26.336	Tetratetracontane	$C_{43}H_{88}$	87	1.72
5	29.289	2-propyl tetradecyl ester	$C_{17}H_{36}O_3S$	86	37.42
6	29.778	Eicosane	$C_{20}H_{42}$	93	7.94
7	36.865	Bis(2-ethylhexyl) ester	$C_{22}H_{42}O_4$	53	50.25

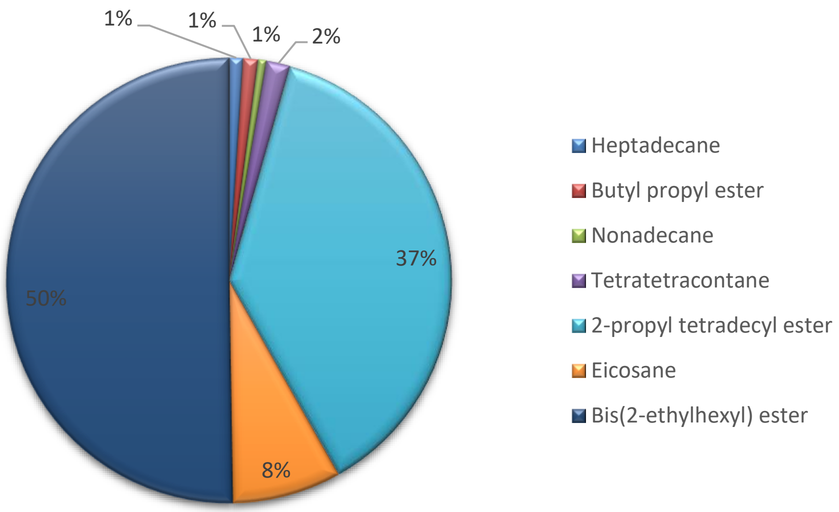

Fig. 2. Component of the MR Fluid (LORD MRF-122EG) obtained by GCMS on HP-5 column

Table 3
Relative concentrations of several classes of volatile compounds in the LORD MRF-122EG fluid

Chemical class of volatile compound	Relative area (%)
Alkane hydrocarbons	11.25
Mineral acid	37.42
Organic acid	51.33

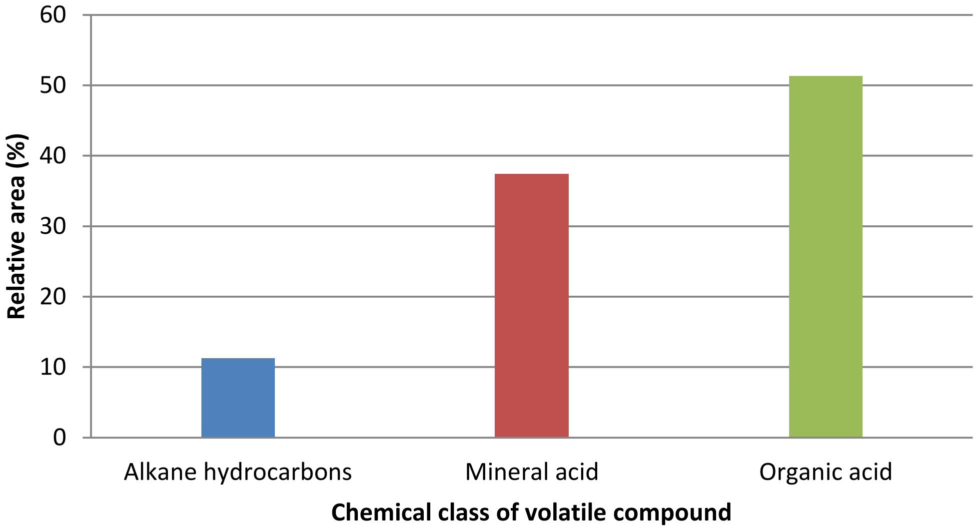

Fig. 3. Relative concentrations of several classes of volatile compounds in the LORD MRF-122EG fluid

Although mineral acid and organic acid have been identified as the dominant component in the carrier fluid, the authors believe the alkane hydrocarbons group contribute to the oily-like properties of the carrier fluid. Alkanes hydrocarbons from hexadecane (C16) upwards form the most important components of fuel oil and lubricating oil [14,15].

In this study, Eicosane was found provided the largest percentage of significant clusters of alkane hydrocarbon. Eicosane has 366,319 constitutional isomers. Despite its high number of isomers, Eicosane suffers from lack of use in the petrochemical industry because of its high flash point properties [16,17]. Flash point is defined as the minimum temperature needed for liquid to ignite on its surface. Material with a high flash point is difficult to ignite [18]. It is encouraging to compare Eicosane with that found by Shetty and Prasad who found that synthetic oil-based MR fluid possesses the important characteristic of the high flash point so it does not thicken at higher temperature [19]. Another important finding was that a component of mineral oil also detected to be one of the components. Mineral oils are neither biodegradable nor environmentally friendly. The component being describe is Nonadecane which has straight-chain alkane with 19 carbon atoms. Nonadecane is a component of petroleum products and released into the environment through the processing and combustion of petroleum products containing this chemical [20]. The value of Heptadecane and Tetratetracontane suggest that a weak concentration of essential oil-based plant may exist in the mixture. Tetratetracontane is a long chain alkane. It is the main components of essential oils from Euphorbia macroclada [21]. On the other hand, Heptadecane is used as an internal standard in the reaction to study the first direct comparison of kava lactone extraction efficiencies and as the appropriate suspended solvent for the extraction and concentration of essential oil [22].

The high presence of 2-propyl tetradecyl also generally known as the sulphurous acid in the essential carrier fluid are believed to act as additive or surfactants for the mixture. The investigation of sulphurous acid was first undertaken by Schroeter. He reported that sulphurous acid gives the desired anti-oxidant protection in the pharmaceutical industry [23]. By drawing on the concept of sulphurous acid anti-oxidant properties, the authors are certain it plays the same role in the mixture of the MR fluid. Several patents of MR fluid from LORD Corporation has mentioned the use of anti-oxidant to improve its durability [24,25]. It is also interesting to note the existing of organic acid dominating the volume percentage of the mixture. The organic acid detected were Butyl propyl ester and Bis(2-ethylhexyl) ester. The observed difference between Butyl propyl ester and Bis(2-ethylhexyl) ester in this study was too significant. Butyl propyl ester was further categorized as fumaric acid are usually used as food additives because of its hydrophobic nature. There are similarities between the nature expressed by fumaric acid and those fumed silicas described by Alves *et al.,*. Even though, it less pronounced in the mixture (1.08%), the authors believe fumaric acid as an agent to enhanced thixotropic effect to the overall MR fluid stability and accordance to the previous study [26].

One unanticipated finding was that Bis(2-ethylhexyl) ester (a decanedoic acid or commonly known as Sebacic acid) is a dicarboxylic acid and is a derivative of castor oil [27]. Castor oil is a vegetable oil obtained by pressing the seeds of the castor oil plant (Ricinus communis) [28,29]. This finding further supports the idea of the MR fluid was actually formulated by taking into account the cost and environmental issue [30]. However, it is important to bear in mind the possible bias in these finding such that the probability was 53% only. These finding, therefore, need to be further interpreted with caution. A further study with more focus on the retention time of 36.865 minutes with the probable standard solution is therefore recommended.

4. Conclusion

The main goal of the current study set out to identify the component of the carrier fluid from a vehicle smart fluid which is an original equipment manufacturer (OEM) MR fluid. The study has shown seven components were found. One of the most significant findings to emerge from this study is that Bis(2-ethylhexyl) ester was found dominating the concentration of the mixture. However, this finding did not have a high confirmation probability. Whilst this study did not confirm the high presence of oil, it did partially substantiate the presence of alkane hydrocarbons that formulated the oil. The second major finding was the existence of two additives. Taken together, their existence suggests that more than one additive was added into the mixture to stabilise the MR fluid. This research extends our knowledge of the actual formulation of MR fluid may comprise many components not limited to just one carrier fluids and one additive. The generalisability of these results is subject to certain limitations. For instance, there is no internal standard were applied. The authors believe that the seven major components in the essential carrier fluid of MR fluid detected should not be ignored, as they also strongly contribute to the overall qualities of the MR fluid for vehicle industrial application.

Acknowledgement

The authors wish to thank Malaysia Ministry of Higher Education and Universiti Teknologi Malaysia for financial support under Tier-1 Research University Grant (GUP) Project Vot No. Q.K130000.2543.19H85.

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