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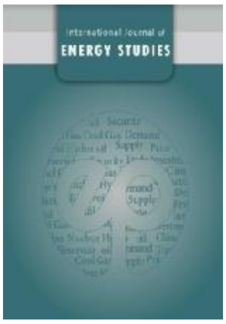

Research Article

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Experimental investigation of the effects of biogas source H_2SO_4 on engine oil and bearing materials in gas engines

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Highlights

- Investigation of H_2SO_4 effect on materials in combustion
- Investigation of the effect of H_2S formation in materials in combustion
- Investigation of the effects of combustion gases on the wearing amount

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ABSTRACT

In this study, as a result of the combustion reaction of H_2S in the content of biogas, its effects on engine oils, engine bearings and bearing materials were evaluated by transforming into H_2SO_4.Then, four ball tests were performed by adding 1% H_2SO_4 to four kinds of gas engine oils. As a result of the tests, it has been determined that H_2S in the content of biogas has adverse effects on lubrication. Oils that have lost their lubrication feature have been found to increase the wear on metal parts by 50-70% on average. In addition to these results, copper rod test was carried out on the same oil samples. Monthly H_2S values were followed in a biogas plant used in energy production from waste. Changes in turbocharged bearings were observed after using of variable H_2S ratio of 750 and 1500 hours. These changes were compared with field studies in the literature. It has been determined that the disruptive / corrosive effect resulting from the oxidation of oil or mixing with acid in any way is very important.

Keywords: *Lubrication oil, biogas, four ball test, H_2S, H_2SO_4*

1. INTRODUCTION

Biogas is the organic conversion of carbon dioxide and methane gas under the influence of microbiological flora in an anaerobic environment. There may be varying amounts of hydrogen sulfide, sulfur dioxide, water vapor, nitrogen, and hydrogen gas depending on climatic differences, the size of the digester plant, and the raw material [1,2]. Biogas, a renewable energy source based on biomass, is used in both internal combustion engines and in energy conversion by providing direct combustion. During the process, it is not efficient to compress biogas at high pressures in steel tubes, as in compressed natural gas (CNG). For this reason, the process is completed by injecting the biogas directly into the internal combustion engine at a certain pressure after leaving the reactor [3,4].

Abrasion is metal losses on the surfaces that move on top of each other as a result of changing the space between the surfaces. Therefore, deterioration of the material is inevitable as a result of this situation becoming permanent. Metal-metal contact is tried to be prevented by adding film-forming agents to the oil between the surfaces. These materials can protect metal surfaces by absorption or chemical reaction [5,6]. Mineral oils are constantly exposed to external influences during storage and usage. The most important result of these effects in oils is the acidification of the oil by providing oxygen to the molecules consisting of carbon and hydrogen atoms. For this reason, manufacturers use various additives consisting of antioxidants, anti-corrosion agents, corrosion inhibitors, cleaners, viscosity regulators and alkaline materials to prevent these deteriorations in oil. Base-based alkali additives are expressed as the "Total Base Number (TBN)" of the oil, but it can be also expressed as an indicator of the oil's ability to absorb acids [7-9]. Although this number varies depending on the amount of the ash in the oil, it is generally between 4.0-14.0. With the use of it, the number of TBN will gradually decrease and the number of TAN will increase. The high TBN value provides protection in the engine against oxidation and abrasion in fuels where acid compounds arise as a result of combustion. However, TBN is not the only factor to consider when determining how long oil is used.

H_2SO_4 in engine oil occurs due to the presence of sulfur-derived materials in fuel and/or sulfur derivatives in engine oil to react with water vapor. The H_2S contained in the biogas inside the combustion chamber creates SO_2 and water vapor during combustion (Equation 1). With condensation, the steam turns into liquid form. SO_2 and O_2 react because the combustion continues, and form SO_3 (Equation 2). Finally, SO_3,which is formed rapidly during combustion, and existing water combines with each other and then forms H_2SO_4 (Equation 3). As it can be seen in the equations, the ratio of H_2SO_4 by H_2S reacting cytoquitometrically is 1:1. Therefore, H_2SO_4 occurs as much as H_2S.

$$H_2S + 3/2O_2 \leftrightarrow H_2O + SO_2 \quad (1)$$
$$SO_2 + 1/2O_2 \leftrightarrow SO_3 \quad (2)$$
$$SO_3 + H_2O \leftrightarrow H_2SO_4 \quad (3)$$

The sulfuric acid resulting from above reaction easily forms condensation on the cylinder wall due to its high dew point. In internal combustion engines operating in facilities that do not have gas cleaning processes, corrosion in various parts, abrasion in moving parts, and deterioration in engine oil occur due to the hydrogen sulfide contained in biogas [10]. In studies related to engines, it was stated that sulfuric acid preserves the gas phase, but this condensation occurs on the cylinder walls depending on the temperature of the gases inside the combustion chamber [11-14]. Sulfuric acid dissolves in $H_2SO_4{}^-$, H_3O^+, SO_4^{2-} and sulfuric acid molecules in a two-step reaction water , depending on temperature and amount of concentration.

$$H_2SO_4 + H_2O \leftrightarrow HSO_4^- + H_3O^+ \quad (4)$$
$$HSO_4^- + H_2O \leftrightarrow SO_4^{2-} + H_3O^+ \quad (5)$$

The exothermic reaction of unsaturated hydrocarbons with SO_3 and H_2SO_4 produces hydrocarbon sulfonates and water [15]. It is reported in the samples from engine oils that there are water droplets and carbon particles in the lubricants [13,16,17]. The resulting sulfuric acid is concentrated on the cylinder walls. The higher dew point is, the easier the condensation can be. According to Verhoff's equation, small amounts of SO_3 appear to cause a large increasing in the dew point. In this equation, partial pressures in the gas are mainly used [12-14].

$$T_d = \frac{1000}{[2.276-0.0294 \times \ln(P_{H2O})-0.0858 \times \ln(P_{SO3})+0.0062 \times \ln(P_{H2O} \times P_{SO3})]} \quad (6)$$

Taking into considering this assessment for a typical engine, it is stated that the dew point is substantially below the in-cylinder gas temperature, and therefore there is no condensation in the gas volume. However, dew point temperatures are well above the cylinder wall, so condensation occurs especially on walls [13,18]. The liquids stripped through piston oil rings are lowered into the sump; on the other hand, those that can not be stripped can cause corrosion on the cylinder walls. The sulfuric produced by fuels with high sulfur content such as biogas as a result of combustion causes the oil to acidify and the viscosity to deteriorate. This acidification causes the viscosity to rise, forming deposits and resin in the engine oil, and reduce lubrication performance [8,9]. Continuous change in the amount of H_2S in the content of the product obtained from the biogas reactor in gas engines and dual-fuel engines, where biogas is used as the main fuel, makes it difficult to predict the negative effects. Although it is possible to remove H_2S with various desulphurization methods, it is thought that the effects on oil and bearing materials and the production facilities in the field should be examined together.

Various methods are used to detect the deterioration in engine oils and the changes in their lubrication properties. The most common of these methods is the four-ball test method. In this study, 1% H_2SO_4 was added to the oils of different brands and features used in a 61-liter gas engine. In this way, it is aimed to detect the deterioration in the oils used in the engine due to the conversion of H_2S to H_2SO_4 as a result of the burning reaction in the cylinder. The effects of variable H_2S amount on engine turbochargers in a biogas plant were also investigated depending on the time. Biogas contains H_2S in addition to CH_4, CO_2 and other gases. In internal combustion engines, H_2S turns into H_2SO_4 during combustion. H_2SO_4, which is formed as a result of combustion, condenses by adhering to the lubricating oil on the cylinder walls. Condensed H_2SO_4 falls into the crankcase together with the oil and spoils the entire engine oil. This deterioration in the oil causes shortening of the oil life, increasing the friction on the parts, and causing corrosion on the parts. On the basis of the study, the resistance of different oil samples to this acid effect was investigated with available facilities.

2. MATERIAL AND METHOD

In this experiment, 1% H_2SO_4 was added to the oil in four different brands and properties and subjected to degradation. Afterwards, these oil samples were tested in the OPET FUCHS (İzmir) accredited laboratory. In the tests, the differences between the original of the same oil and the state after adding H_2SO_4. Four ball tests were carried out using the ASTM D 4172 test method. In this test, three balls made of stainless steel are located at the bottom and one ball is located at the top basically (Figure 1). The anti-wear properties of the fluids to be tested are determined using a microscope. Apart from this, the distortion diameter and volumetric calculations occurring on the ball surface are also used [19].

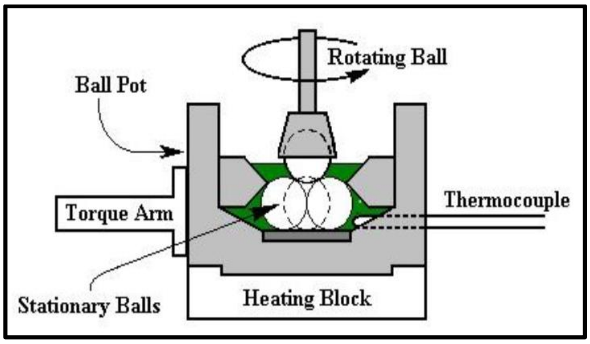


Figure 1. Four ball test

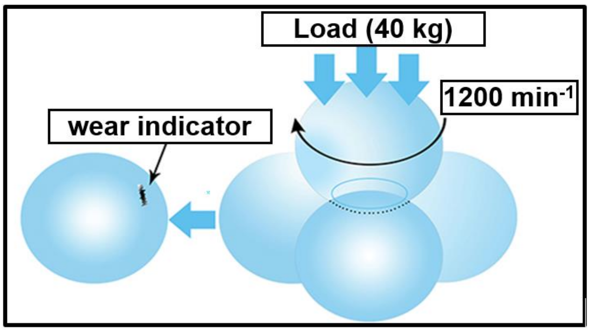


Figure 2. Principle of four ball test

In the four-ball tests, the ball at the top was rotated at the 392 N load and a speed of 1200 min^{-1} and the temperatures of the fluid lubricant samples were kept constant at 75 $^{\circ}$C. The test duration was determined as 60 minutes. This test was repeated for all oil samples. It has been used 8 samples with 4 original oil samples and with added 1% acid in the experiments. Before starting the tests, all test balls were thoroughly cleaned with toluene and dried with hot air. As shown in Figure 2, events such as deterioration and wear on the surfaces of the bottom balls were observed and the results were examined. Each of the oil samples belongs to different companies. Oil brands and models were kept confidential because it was evaluated that this study in the academic field was compared with the results of the experiments and the company could not comply with the ethical rules of the companies (Table 1-4).

Table 1. Features of 1st Oil sample

Oil Feature	**Values**
SAE	15 W – 40
Kinematic Viscosity @ 40 ^{o}C mm^2/s, ASTM D445	114
Kinematic Viscosity @ 100 ^{o}C mm^2/s, ASTM D445	15,0
Viscosity index, ASTM D 2270	137
Sulphated ash, Mass Fraction ASTM D 874	0,98
Total Base Number mgKOH/g, ASTM D 2896	10,4
Yield Point ^{o}C, ASTM D 97	-30

Flash Point°C, ASTM D 92	236
Intensity @ 15 °C kg/l, ASTM D 4052	0,876

Table 2. Features of 2nd Oil sample

Oil Feature	**Values**
SAE Vicosity Degree	40
Kinematic Viscosity @ 40 °C mm^2/s, ASTM D445	125
Kinematic Viscosity @ 100 °C mm^2/s, ASTM D445	13,5
Intensity @ 15 °C, kg/l, ASTM D4052	0,890
Flash Point (Cleveland Open Cup Method), °C, ASTM D92	268
Yield Point °C, ISO 3016	-18
Total Base Number mgKOH/g, ASTM D 2896	5,3
Sulphated Ash% wt, ISO 3987	0,57
Phosphor ppm, ASTM D4047	300

Table 3. Features of 3rd Oil sample

Oil Feature	**Values**
SAE Vicosity Degree	40
Kinematic Viscosity @ 40 °C mm^2/s, ASTM D445	145
Kinematic Viscosity @ 100 °C mm^2/s, ASTM D445	14,5
Viscosity index ASTM D2270	98
Intensity @ 35 °C, kg/l, ASTM D4052	0,896
Flash Point (Clevland açık kap metodu), °C, ASTM D92	272
Yield Point °C, ASTM D97	-21
Total Base Number mgKOH/g, ASTM D 2896	5
Sulphated Ash % wt, ISO 3987	< 0,5
Phosphor ppm, ASTM D4047	~ 300

Table 4. Features of 4th Oil sample

Oil Feature	**Values**
SAE Vicosity Degree	40
Kinematic Viscosity @ 40 °C mm^2/s, ASTM D445	-
Kinematic Viscosity @ 100 °C mm^2/s, ASTM D445	13,3
Viscosity index ASTM D445	70
Intensity @ 20 °C, kg/l, ASTM D1298	0,825-0,850
Flash Point °C	182
Yield Point °C	-18

The aromatic and paraffinic oil ratios in the oils were also examined by conducting a copper rod experiment. After the suspension mechanism was prepared, some of the rods were inside the oil and some were out of the oil, they were kept at 150 °C for 24 hours.

Copper rod corrosion test is a test for determining the corrosive effect of engine oils on metals. This test is carried out in accordance with ASTM D 130 test standard. This standard covers all petroleum-derived fuels (land, air and sea vehicles), solvents, mechanical oils and hydrocarbons with pressures not greater than 124 kPa at 37.8 °C [20]. For the determination of the corrosive effect, a change in the copper bar was observed as a result of keeping the copper rod in the oil at a certain temperature and for a certain period of time. The copper bar should be cleaned with fine sandpaper before the test process and its colour should be revealed. As the corrosive effect of the oil increases, the colour of the copper bar becomes darker. Accordingly, the colour changes formed on the copper bar after it was kept for 24 hours at 150 °C in oil samples, were compared with the previously standardized colour changes scale. The standard scale of this colour change can be seen in Table 5.

Table 5. Definitons of Colour Scale of Copper Strip Corrosion (ASTM D130)

Classification	Indication	Definition
1	Slight Tarnish	1a. Light orange, almost the same as a freshly polished strip
		1b. Dark orange
2	Modarete Tarnish	2a. Claret red
		2b. Lavander
		2c. Multi-colored with lavender blue and/or silver overlaid on claret red
		2d. Silvery
		2e. Brassy or gold
3	Dark Tarnish	3a. Magenta overcast on brassy strip
		3b. Multi-colored with red and green showing (peacock), but no gray
4	Corrosion	4a. Transparent black, dark gray or Brown with peacock green barely showing
		4b. Graphite or lusterless black
		4c. Glassy or jet black

3. RESULTS AND DISCUSSION

As a result of four-ball tests applied to oil samples of four different brands designed for engines, original and acid-added data were recorded and the amount of erosion was shown in Table 6 and Figure 3.

Table 6. Abrasion Values of Four Ball Test

1st Sample		2nd Sample		3rd Sample		4th Sample	
Wear on acid-free oil	Wear on oil exposed to acid	Wear on acid-free oil	Wear on oil exposed to acid	Wear on acid-free oil	Wear on oil exposed to acid	Wear on acid-free oil	Wear on oil exposed to acid
522 µm	830 µm	465 µm	797 µm	522 µm	830 µm	465 µm	797 µm
difference: 308 µm		difference: 332 µm		difference: 352 µm		difference: 284 µm	

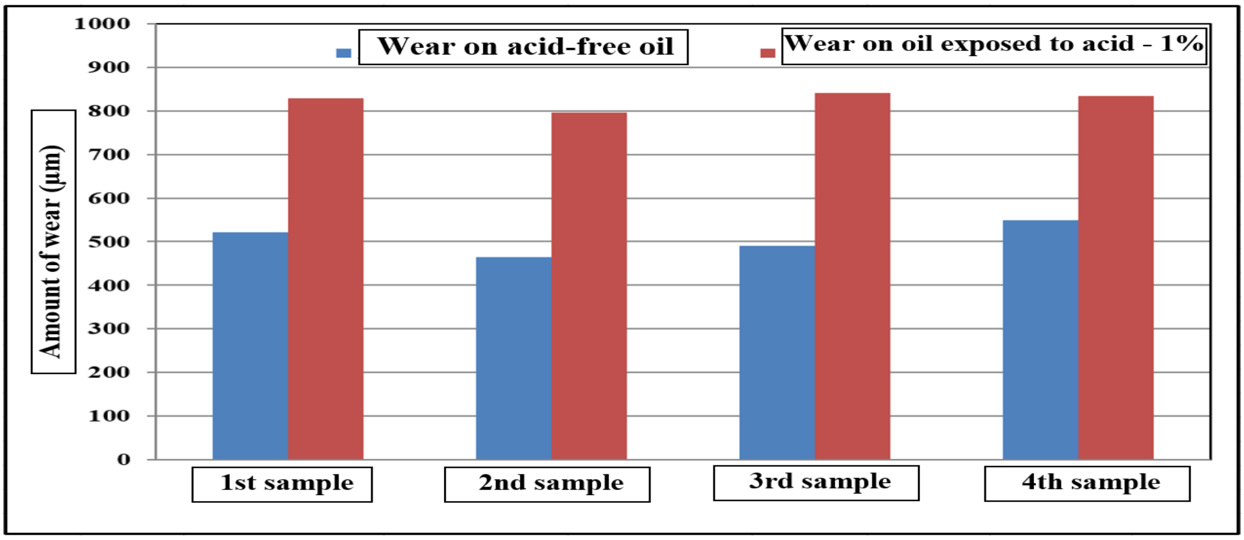


Figure 3. Effects of sulfuric acid on oil samples

As a result of the tests, the normal wear amount was measured as 522 µm in the first sample number 1 which was not subjected to the deterioration, however, this amount was 830 µm after 1% deterioration. The amount of ball abrasion, which was 456 µm before the addition of acid in second oil sample. Then it reached 797 µm after acid addition. After that, it increased from 490 µm to 842 µm in third oil sample and from 550 µm to 834 µm in fourth oil sample, respectively. In terms of the increase in wear rates, 59% in sample 1, 71.4 % in sample 2, 71.8% in sample 3, and 51.6% in sample 4 were obtained. From this point of view, it is seen that oils 2 and 3 lose their lubrication/anti-corrosion properties more quickly due to the effect of H_2S in the biogas, and thus will cause rapid wear in critical engine parts. It has been determined that 1% H_2SO_4 accumulation that may occur in the engine oil as a result of the use of biogas can cause the oil to deteriorate by 71%.

The first three samples consist of gas engine oils in the market; on the other hand, "White oil" is used for fourth sample. Despite not containing any additive packages, it is noteworthy that it contains similar abrasion rates with the first three samples and it shows similar performance after the addition of H_2SO_4. It has been evaluated that its content consists of entirely paraffinic oils and resists deterioration.

There is a tendency towards acidification in the nature of the oil, it can be seen that H_2S can degrade the alkali-based additives in the oil very quickly by turning into H_2SO_4 in the combustion reaction. As a result of this deterioration, it was revealed that the oil lost its lubrication and anti-corrosion properties. In engines where biogas is used, it is seen that the oil to be preferred is very important in determining the engine life and sustainability of the system, and trace the amounts of H_2S contained in the gas will affect the engine oil first and then the critical parts of the engine. The changes caused by the deteriorated oil in copper rods are shown in Figure 4. It is aimed to obtain information about the sulfur and aromatic oil ratios in the oil samples used in this test. Visual distortion and cracking effects occurring in the bars after the test is completed are shown in Figure 4 and the color scale is shown in Figure 5.

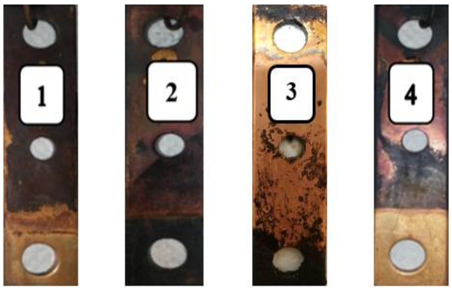


Figure 4. Copper rod samples

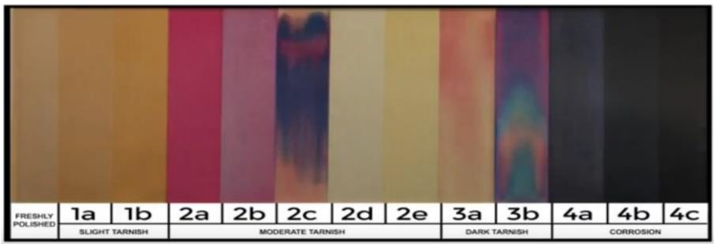


Figure 5. Colour scale

Accordingly, there was no significant physical change in the part of the rod in the oil which was found in oil number 1; on the other hand, it was observed that there was a significant amount of corrosive effect in the region outside the oil. In a similar way, it was observed that the stick in oil number 4 was affected, but this effect was less. On the other hand, the stick in 2nd oil sample was completely affected. In 3rd oil sample, it was determined that the coppers in 1st oil sample and 4th were in the dark orange color (1b) in the lower parts (in oil), and in the upper parts contacting the oil vapor it was colored graphite or matt black (4b). It was determined that corrosion in the form of dark black and graphite (4b and 4c) occurred in the entire rod of 2nd oil sample. On the copper rod immersed in the oil sample number 3, it was observed that the part of the oil remained dark and corroded (4a).

It is considered that the exposure of the parts other than the oil in the 1,2 and 4 rods to a faster deterioration compared to the parts of the oil is caused by the evaporation of the sulfur compound in the base oil under high temperature to cause oxidation. In this experiment, it is thought that the parts under the oil are caused by sulfur and aromatic compounds depending on the base oil, oxidation due to cracking and oxidation effects, and the parts above the oil due to evaporating sulfur. In the experiments, the effects of the resistance differences of the oils introduced in the market in accordance with the use of biogas and the quality of the base oil have been observed. Accordingly, in a situation that the amount of H_2S in biogas is at a low value, it significantly shortens the life of the engine oil. Therefore, it is going to be required to determine useful life for the new situation by testing the engine oils in advance in the case of biogas use. Otherwise, it is considered that the deterioration in the oil first will cause serious damage in the engine bearings and other parts of the engine.

After these laboratory tests, turbo bearings of 1 MWe biogas engine, which is powered by biogas and produced by TEKSAN Generator company, were examined. The H_2S values contained in the biogas used as fuel in this engine are shown in Figure 6. As seen in real usage conditions, this value changes daily depending on many factors such as waste content, fermentation process.

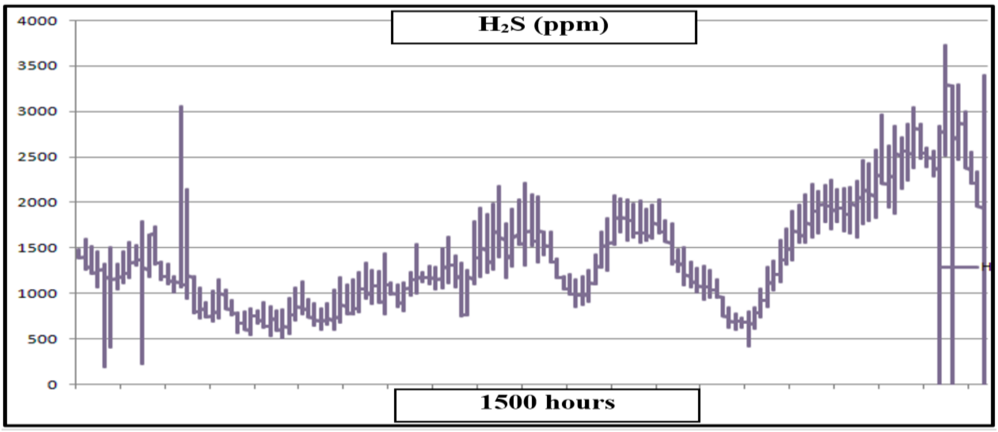


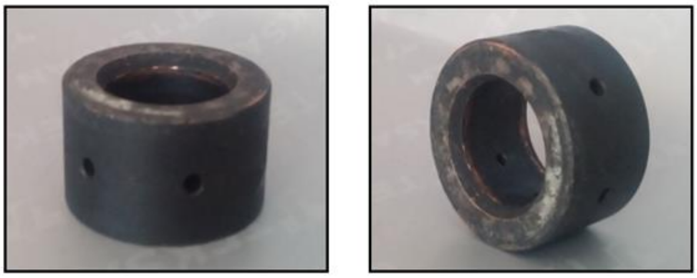
Figure 6. Change of H_2S for 1500 hours

It is observed that there is a continuous fluctuating production of H_2S at an average of 500-3000 ppm within two months. This variable situation is required to be taken into account in engine maintenance and oil change.

Accordingly, the images of 750 and 1500 hours of use were examined after the turbo bearings of the engine were reset. Corrosion and deterioration effects occurring in the bearings are shown in Figure 7 (a) (b), (c).

(a) Appearance of original Turbo Bearings

(b) Appearance of Turbo Bearings after 750 hours of operation



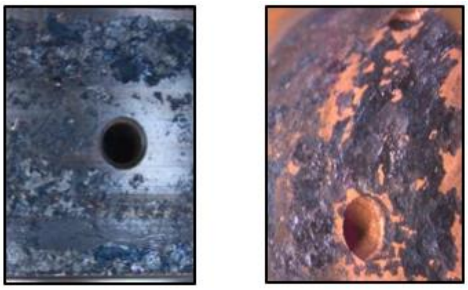
(c) Appearance of Turbo Bearings after **1500** hours of operation

Figure 7. Appearance of Turbo Bearings after use

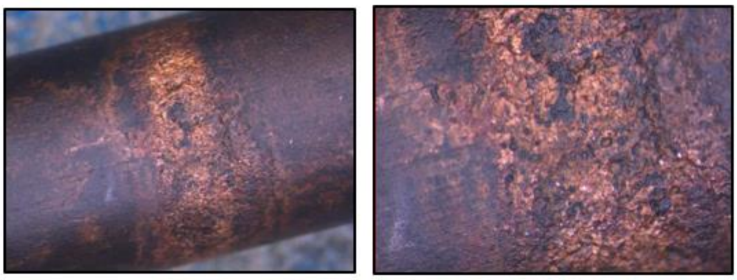
The original image of turbo bearing materials used in the engine is presented in Figure 7(a). In the image (b), a visible change in bed is observed as a result of 750 hours of usage. Finally, in the image (c), the final deterioration in the bearing was shown as a result of 1500 hours of use. This deterioration of the bearings occurs as a result of the chemical reaction of acids with oxides of the bearing metal. It also shows that the disruptive/corrosive effect resulting from oxidizing the oil over time or mixing the acid in any way is also noteworthy. Corrosion inhibitors in oil neutralized light corrosion sources such as incomplete combustion products thanks to the productive film layer formed on metal surfaces. However, the corrosive effect of water vapor can be vital with the oil deteriorating primarily and the inhibitors being deactivated, as the bearings become susceptible to all kinds of harmful effects [21-23]. It is considered that this situation will cause many parts with a much longer service life to be taken to maintenance in a short time and as a result of time and money loss.

In addition to the corrosive effect, viscosity regulating additives are also used to minimize the effect of engine oils from the temperature difference. Especially, it is expected that the viscosity of multigrade oils over a wide temperature range will not change much and this change will remain within certain limits. These additives are polymer molecules with a long chain structure and a flexible and expanding structure. These flexing and expanding molecules create resistance within the thinned oil and minimize the viscosity loss of the oil [6]. Therefore, in the examination of the traces of friction occurring in the bearings, it was concluded that the acid in the oil broke down this polymer structure and the oil can not maintain its viscosity resistance at high temperatures. H_2SO_4 occurring during combustion in diesel engines, leaks from the combustion chamber to the crankcase between piston rings and cylinder walls. In addition, the oil, which is a thin film layer in the cylinder walls, is also affected by the reactions occurring in the combustion chamber and can be stripped to the sump during the movement of the piston to the BDC (Bottom Dead Center) as some of the burnt gases adhere on it. Apart from this, it is thought that the acid and water vapor contained in the gases leaking to the crankcase can be concentrated in the oil without being removed from the upper crankcase by means of the crankcase ventilation system, thereby causing a rapid deterioration in the oil, due to the low combustion temperature of the biogas.

These bearing materials were compared to the previous field studies[24] by Caterpillar. Visual data related to the previous results are shown in Figure 8. In these tests, corrosion formation was observed in turbochargers (a) and engine oil coolers (b).



(a)Inspection by the (a)Caterpillar-Turbo charger bearing(Cat. 2015).



(b)Inspection by the (a)Caterpillar-Oil Cooler (Cat.2015)

Figure 8. The results of Field Studies

Corrosion occurring in the bearing and oil cooler exchangers were in a similar way. It is thought that this situation will result in both changing the bearings and the oil cooler in the upcoming time.

4. CONCLUSION

Consequently, H_2S turns into H_2SO_4 during combustion in internal combustion engines. H_2SO_4 is formed as a result of combustion and condenses by adhering to the lubricating oil on the cylinder walls. Condensed H_2SO_4 falls into the crankcase together with the oil and spoils the entire engine oil. This deterioration in the oil causes shortening of the oil life, increasing the friction on the parts, and causing corrosion on the parts. The resistance of different oil samples with effect of this acid has been investigated with available facilities on the basis of the study. In the tests made with oil samples that simulate the effects of H_2S in the content of biogas, it was determined that there were adverse effects on lubricants. The oils having lost their lubrication feature, were found to increase the abrasion in the metal parts by 50-70% on average depending on the period of use. This situation causes a shorter oil change period. The H_2S in biogas as the main fuel turns into H_2SO_4 as a result of the combustion reaction. This acidic structure adheres to the oil in the cylinder walls and is stripped to the crankcase by the rings during the movement of the piston BDC. In addition, acid vapor in the gases escaping from the piston ring set to the piston bottom area by compression leaks condenses and affects the oil in the crankcase. The positive crankcase ventilation system in the engines ensures that these non-condensable gases are re-burned by giving them back to the suction system. However, this situation causes the acid vapor to be repeatedly burned in the combustion reaction in the engines that will work with raw gas. In the recurrence of these reactions in each cycle causes the cumulatively increasing amount of acid deposits to occupy the engine oil and lose the lubricating properties of the oil.

As a result of these studies, it is seen that it is important to remove the H_2S in its content or to reduce it to the possible lowest level before the use of biogas in engines. It is considered that taking additional measures to routinely control the H_2S occurring in the biogas reactor as a raw material for the digestion of waste and to obtain the final products and to remain at very low ppm levels will be effective in extending the life of the engine and reducing the oil costs. The positive crankcase ventilation mechanism in the engines to be used for this purpose is transferred to the area like an external tank and thanks to this method the acid amount can be reduced by condensing the acid vapor and sending the remaining gas mixtures to the engine.

In addition to these experiments carried out under laboratory conditions, the biogas-powered cogeneration engines used in the field were also examined. Accordingly, as a result of 1500 hours of operation, it was observed that the H_2S in the biogas content used daily fluctuates constantly due to the content of the waste and can not be kept at a fixed value due to the inadequate measures taken. Even in the long-term operation in which the engine oil is changed regularly, deteriorations were observed in the engine bearings. It was also expressed visually that turbo bearings and oil cooler units were affected. Therefore, it is necessary to redesign the bearing and other important parts of the biogas engine and to choose different bearing materials with very low or low copper content.

The reproduction of oil and material studies is an important step in considering biogas as a sustainable resource. The innovations emerging as a result of these efforts will support the rapid increase in the number of small-scale enterprises.

As a result, all tests and calculations show that the high H_2S value will be the biggest obstacle to maintaining all the benefits of biogas. It was seen that purifying H_2S by various methods before using biogas as fuel will contribute greatly to the whole system.

Scientific infrastructure needs to be created by increasing the number of similar studies in different fields such as energy costs, vibration values, noise emissions, oil analysis. Particularly, it is evaluated that the provision of incentives by the government / private institutions or the inclusion of existing supports in the field of applications of similar studies based on biofuels can accelerate the use of alternative fuels. As a consequence, it is vital to eliminate H_2S and H_2SO_4, which cause corrosion in internal combustion engines using biofuels in the defense industry.

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Declaration of Ethical Standards

The authors of the paper submitted declare that nothing which is necessary for achieving the paper requires ethical committee and/or legal-special permissions.

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