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EFFECT OF ADDITIONAL CO_2 AND N_2 GAS BLANKET TO REDUCE BIODIESEL DEGRADATION IN STORAGE TANK Dedy Wahyudi1, Mas Fawzi2, Beny Cahyono3, Siti Yuniarti4 1Naval Architecture, University Muhammadiyah Surabaya, Indonesia, dedy.wahyudi@ft.um- surabaya.ac.id; 2Mechanical Engineering, Universiti Tun Hussein Onn Malaysia, Malaysia, fawzi@uthm.edu.my; 3Marine Engineering, Sepuluh Nopember Institute of Technology, Indonesia, cak_beny@yahoo.com; 4SOS FA Laboratory, sitiyuniarti@gmail.com ABSTRACT Ship movement and contaminants may be the main causes of accelerated biodiesel degradation during the ship storage process. One of the techniques to protect biodiesel storage on the ship is by applying a blanketing system. In this study, CO_2, N_2, and natural air blankets were added to the Biodiesel stainless steel tanks (B100 and B50) that swings in sinusoidal movement to simulate ship movement at sea and stored for six months. It was found that storing biodiesel B100 and B50 in a blanket with natural air, CO_2, and N_2 gases for 210 days has changed the characteristics of biodiesel in proportional to the storage duration. The addition of CO_2 gas was found to reduce the oxidation effect compared to other blanket gases, but it increases the particle count, TAN, flash point, and density. I n d e x T e r m s : Biodiesel, Storage, Degradation Gasses, Characteristic 1. INTRODUCTION The Indonesian government has obliged that all industries, automobiles and the marine sector has been using palm oil biodiesel (B30) since 2019. Biodiesel or FAME (Fatty Acid Methyl Ester) is obtained from the esterification and transesterification process. Biodiesel is an alternative fuel without aromatic and sulfur content that is easy to degrade and does not cause significant air pollution [1]. During the bunkering process, biodiesel is filled into ship storage tanks, normally in the land storage tank, biodiesel must be stable for at least 3 months. Some research has found that biofuel degradation can be extended up to 6 months with the addition of antioxidants depending on biofuel quality and characteristics [2]. Biodiesel implementation of the ship is still experiencing several obstacles, especially the decrease in power, blocking filters, and damage to spare parts, especially in the fuel system which is faster caused by biodiesel degradation. Some of the causes of degradation in biodiesel are due to the Solvency Effect where ester compounds. Accumulation of water at the bottom of the tank will encourage the growth of many microbes. The difference in affinity levels of biodiesel and diesel oil causes the formation of an emulsion which is characterized by the cloudiness of the fuel. Water content in the biodiesel is also caused by the growth of fungi and bacteria. Microbiology that grows in biodiesel causes blocking conditions on pipes and filters of the engine. The hydrolytic reaction begins with a high content of free fatty acids and water, as well as other hygroscopic impurities[3]. Biodiesel degradation during storage also can be caused by hydrolytic and oxidative reactions. In the early stages, the formation of free carboxylic acids is characterized by an increase in the acid number, then hydro-peroxides are formed, followed by the formation of polymer products and an increase in the viscosity of the fuel. The hydrolytic reaction begins with a high content of free fatty acids and water, as well as other hygroscopic impurities. The rate of oxidative degradation depends on the composition of free fatty acids, exposure to air, light/sun heat, and antioxidants [4]. The main cause of biodiesel degradation is due to the air contamination and condensation of water that enters the storage tank, Air contains several particles including 78.1% Nitrogen (N_2), 21% Oxygen (O_2) and 0.9% are containing Ar, Ne, He, H_2O, CO_2, and others. Several articles mentioned that biodiesel degradation can be reduced with a nitrogen blanket in the storage tank, but it is still not known with certainty the efficiency of nitrogen administration on the quality of biodiesel. Storage of the fuel system on ships still used natural air vents, where air can enter and leave freely[5]. The gas blanket process generally was carried out for fire protection, especially for tanker ships. Gases are used CO_2 for oil tankers and N_2 for chemical tankers. Most oil tankers use CO_2 because the process is more economical. It can be produced from the combustion system obtained by the generator and main engine. The basic method of slowing down the oxidation changes of fats is to eliminate biodiesel contact with oxygen. This can be done by replacing oxygen with an inert gas blanket. Nitrogen and carbon dioxide are commonly used in the food industry to reduce the autoxidation process. Nitrogen is poorly soluble in both water and fat. For this reason, it is used as a fill gas, which reduces the concentration of other gases in the package and prevents it from collapsing. Carbon dioxide is highly soluble in both water and fat, and its solubility increases with decreasing temperature. Carbon dioxide is more permeable through the package than nitrogen [6]. The density of gas blanket that is generally used, CO_2 gas (44 g/mol) is heavier than Nitrogen (N_2) (28 g/mol) and O_2 (32 g/mol). The objective of this study is to investigate the effect of additional N_2 and CO_2 gas blankets in the storage tank on the physicochemical properties of biodiesel. 2. MATERIAL AND METHOD 2.1. Biodiesel Palm Oil The main source of raw material for biodiesel in Indonesia is using palm oil. The biodiesel production process generally used a methanolysis (transesterification with methanol) reaction, which is a reaction between vegetable oil and methanol assisted by a base catalyst (NaOH, KOH, or sodium methylate) to produce a mixture of fatty acid methyl esters with glycerol as follow-up products[7]. In this study, Biodiesel (B100) samples were produced from palm oil Fatty Acid Methyl Ester (FAME) by PT. Wilmar. Furthermore, the biodiesel B50 made with 50% FAME and 50% Pertamina DEX as fossil oil. 2.2. Gases The Nitrogen (N_2) and Carbon dioxide (CO_2) gases were delivered by PT. Wonokoyo, where the purity of gasses around 99,9% food-grade standard in the high-pressure tank. The gases were distributed to the stainless steel storage tank via a regulator valve to reduce the pressure from 200 bar gas to around 0.2 bar gas. The regulator valve to be used keeps gas pressure in the storage tank if any leakage in the system. The pressure of gas in the storage tank can be monitored by a pressure gauge. 2.3. Biodiesel Storage Condition The sample of biodiesel B100 in this research has been used PT. Wilmar Biodiesel product. One of the biggest biodiesel refineries is located in Gresik City, East Java, Indonesia. The Biodiesel was stored in two types B100 and B50. Biodiesel B100 means 100% Fatty Acid Methyl Ester (FAME), and Biodiesel B50 means 50% FAME was blended with 50% fossil oil. It was stored in 6 units of stainless steel cylinder tank filled with eight liters for each sample of biodiesel, Furthermore, all the tanks were placed on the cradle. An electric motor with 500 rpm was installed to swing the cradle in sinusoidal movement at approximately 7.6 degrees as shown in fig.1. Biodiesels sample was placed in the dark room. The room temperature was maintained at approximately 28oC with an air conditioning system, The air humidity has been maintained not less than 70% with a humidity sensor, water spray nozzles, an electric pump, and two electric fans in the room that works automatically. Fig 1. Biodiesel storage condition Description : A- Pipe & valve connection E- Gearbox for gas blanket F- Pressure gauge B- Stainless Steel Biodiesel G- Holder Storage Tank H- Steel Cradle C- Connecting rod I – Drain valve D- Electric Motor J – Filling Cap J I 2.4. Biodiesel Sampling The samples of biodiesel were stored for 210 days. At every 45-day interval, the samples were taken at about 400 ml. The preparation of sampling from the storage tank has been stirred using a magnetic bar with a length of 5 cm into the storage tank at the beginning of the biodiesel filling. It was done for 60 minutes to make sure the biodiesel was completely mixed. The samples were drained through a drain valve located at the bottom side of the tank. Then a total of 350 ml was filled into a bottle which has a capacity of 1 liter made of HDPE material. Furthermore, biodiesel samples in the bottles were filled with gases blanket and sent to Laboratory. 2.5. Properties Test The Physico-chemical properties test of biodiesel in this study was done according to ASTM and EN standards [2]. Several key properties were measured to determine the quality of biodiesel, they are flash point, kinematic viscosity, density, water content, and particle count 4, 6, and 16 microns [6]. Chemical characteristics were monitored Oxidation stability, Total Acid Numbers (TAN), and FAME content. 3. RESULT AND DISCUSSION 3.1. FAME Content FAME content testing in this study is expected to determine the effect of blanket gas on biodiesel quality by measuring the percentage of FAME in biodiesel. Testing was carried out with Eralytic FTIR 100 FAME content(%) BB5500--NCAO2 80 B50-N2 B100-NA B100-CO2 B100-N2 60 0 50 100 150 200 250 Duration (Days) Fig 2. FAME content of biodiesel blanket. The results of the FAME content test on biodiesel B100 during storage there found a decrease of about 3% in a few days of sampling. As shown in fig. 2, the average value of FAME content of biodiesel B100 CO2, N2, and NA blanket was no significant decrease. FAME content in biodiesel blending B50 was known increased along with the duration stored. Biodiesel bland B50 samples with CO2 blanket had a lower value, while the Biodiesel N2 blankets had a higher value than NA blankets. 3.2. Water Content The water content in biodiesel can be classified as free, emulsion, and soluble water. It has been tested by Karl Fischer ASTM D1744 with a maximum limit on the diesel oil water content is 500 ppm or 0.05% by volume. The results of measurements of water content in biodiesel B100 since the beginning of sample storage have exceeded the maximum value limit of 500 ppm as ASTM D1744 standard. 600 Water content(ppm) 500 B50-NA B50-CO2 B50-N2 B100-NA B100-CO2 B100-N2 400 ASTM D1744 (max) 0 50 100 150 200 250 Duration (Days) Fig 3. water content of biodiesel blanket. As shown in fig. 3 the value of water content in the biodiesel B100 has increased with a longer storage duration. The effect of the Blanketing system shows that the Biodiesel B100 NA blanket was known to have a higher value than the others, while the N2 blanket sample has a lower value than the CO2 blanket. Water content in biodiesel B50 since the beginning of sample storage was below the maximum limit value of the standard. This overall value of the water content in biodiesel B50 was increased along with the duration stored. On days 125 to 175 the water content value had passed the standard. The effect of the blanketing system has been found that the Biodiesel B50 N2 blanket is known to have a higher value than the others, while the CO2 blanket sample has a lower value than the NA blanket. but on days 171 and 210 the water content value of the B50-CO2 blanket was above the B50-NA blanket. 3.3. Total Acid Number Total acid number (TAN) is the mass of potassium hydroxide (KOH) in milligrams, needed to neutralize one gram of a chemical. Standard methods forthe determination of acid numbers were conducted according to ASTM D664 by Titrator 848 Titrino Plus. 0,7 Total acid number (mgKOH/g) 0,6 0,5 0,4 0,3 B50-NA B50-CO2 0,2 B50-N2 B100-NA B100-CO2 0,1 B100-N2 ASTM D664 (max) 0 50 100 150 200 250 Duration (Days) Fig 4. The total acid number of biodiesel blanket. Total acid numbers were measured by the Titration Method. The test results of the TAN value of biodiesel B100 gasses blanket are all still below the ASTM D664 standard. From fig 8. The TAN B100 blanket has an increasing trend with the length of storage time. The effect of the blanketing system shows that Biodiesel B100 Natural Air Blanket has a higher value than the others, while the N2 blanket sample has a lower value than the CO2 blanket. The measurement results of the TAN value of biodiesel B50 gas blanket were all still below the standard except for CO2 blanket on day 171 which was higher. The TAN values of B50 blanket has also increased along with the storage time. The effect of the blanketing system was found biodiesel B50 CO_2 blanket have a higher value than the others, while the N_2 blanket sample has a lower value than the NA blanket. 3.4. Oxidation Stability Oxidation Stability is the tendency of fuels to react with oxygen at ambient temperatures and describes the relative susceptibility of the fuel to degradation by oxidation. In this study, the measurement of oxidation stability was carried out by Rancimat EN15751 standard. 35 B50-NA B50-CO2 30 B50-N2 B100-NA B100-CO2 Oxidation stability (hr) 25 B100-N2 EN15751 (max) 20 15 10 5 0 0 50 100 150 200 250 Duration (Days) Fig 5. Oxidation stability of biodiesel blanket. The results of the oxidation value of the biodiesel B100 gas blanket were initially above the standard 3 hours. In Fig.5 shows that the oxidation stability value decreased along with the longer storage time. The effect of the blanketing system shows that the Biodiesel B100 CO_2 blanket was found to have a higher value than the others, while the NA blanket sample has a lower value than the N_2 blanket. The biodiesel B50 gasses blanket as shown in fig. 5. In the beginning of the storage process until day 85 there was a significant decrease, but after that, the oxidation stability values doesn't seem to decrease significantly. The biodiesel, B50 with CO_2 blanket was known to have a higher value than the others, while the biodiesel with NA Blanket has a lower value than the N_2 Blanket. 3.5. Flash Point Flashpoint is the temperature at which the fraction will evaporate and cause fire when exposed to sparks or high temperatures and pressures. This characteristic was determined by ASTM D93 method. The Flash point number for minimum marine fuel according to IMO regulation was mentioned as not less than 60℃ [8]. The result of measuring the flash point value of the B100 blanket overall is above the ASTM standard also Marine fuel standard as shown in fig.6. The results of testing the flash point value of the overall B50 gas blanket are above ASTM D93 standard also marine fuel standard. The flash point value has increased with the duration of biodiesel storage. 180 160 Flash point (oC) 140 120 100 80 60 B50-NA B50-CO2 B50-N2 B100-NA B100-CO2 B100-N2 D93 (min) ISO8217 (min) 0 50 100 150 200 250 Duration (Days) Fig 6. Flash point of biodiesel blanket. The effect of the blanketing system shows that the biodiesel CO_2 Blanket was found to have a higher value than the others. The biodiesel with natural air blanket sample has a lower value than the N_2 blanket. 3.6. Density Density is a comparison between two principal quantities, namely mass, and volume. The specific gravity of diesel fuel is measured using the ASTM D1480 method at a temperature of 15℃. As shown in fig.7 Biodiesel density from day to day obtained a fluctuating value, the whole sample showed an increase but it was still in the standard values. 910 900 890 880 Density (kg/m3) 870 860 B50-NA B50-CO2 850 B50-N2 B100-NA 840 B100-CO2 B100-N2 830 EN1414 min EN1414 max 0 50 100 150 200 250 Duration (Days) Fig 7. The density of biodiesel gasses blanket. The density value of the B100 blanket has slightly increased along with the storage time of biodiesel. The effect of the Blanketing system shows that the Biodiesel B100 CO_2 Blanket was found to have a higher value than the others. The biodiesel with natural air (NA) blanket sample has a lower value than the N_2 Blanket, but after 135 days the N_2 Blanket has a lower density value than the NA blanket. The density value of the B50 blanket has increased along with the storage time of biodiesel. The effect of the Blanketing system shows that the Biodiesel B50 CO_2 Blanket was found to have a higher value than the others, while the N_2 Blanket has a lower density value than the NA blanket. 3.7. Particle Count Particle count is a comparison between two principal quantities, namely mass, and volume. Due to the variety of shapes and sizes of contaminants, In this study was conducted the fuel cleanliness measurement according to ASTM D7596 [9], It has been measured the number of particles larger than 4 μm, 6 μm, and 14 μm in a 100 ml of liquid sample. 20000 B50-NA 18000 B50-CO2 Particle count @4micron (part) 16000 B50-N2 B100-NA 14000 B100-CO2 B100-N2 12000 ASTM D7596 (max) 10000 8000 6000 4000 2000 0 0 50 100 150 200 250 Duration (Days) Fig 8. Particle count 4 μm of biodiesel blanket. 10000 B50-NA B50-CO2 8000 B50-N2 Particle count @6micron(part) B100-NA B100-CO2 6000 B100-N2 ASTM D7596 (max) 4000 2000 0 0 50 100 150 200 250 Duration (Days) Fig 9. Particle count 6 μm of biodiesel blanket. 4000 B50-NA B50-CO2 Particle count@14micron (part) B50-N2 B100-NA 3000 B100-CO2 B100-N2 ASTM D7596 (max) 2000 1000 0 0 50 100 150 200 250 Duration (Days) Fig 10. Particle count 14 μm of biodiesel blanket. Based on the test results in Fig. 8 to 10, it was found that most of the particles in the biodiesel blanket value exceed the standard. In biodiesel B100 CO_2 Blanket has a value greater than N_2 and NA, while in Biodiesel B50 N_2 blanket has a higher particle number than the others. In general, both biodiesel B100 and B50 values of particle content have increased along with the storage duration. 3.8. Kinematic Viscosity Fluid viscosity is a unit that states the measure of friction in a fluid. In fuel properties, viscosity affects fuel injection capabilities. The viscosity of samples was measured according to ASTM D445 method with a limit of viscosity values on diesel fuel 2.0 - 4.0 cSt. 4,5 4,0 Viscosity (cSt) 3,5 3,0 ASTM D445 (max) ASTM D445 (min B100-N2 B100-CO2 2,5 B100-NA B50-N2 B50-CO 2 2,0 B50-NA 1,5 0 50 100 150 200 250 Duration (Days) Fig 10. Kinematic viscosity of biodiesel blanket. Biodiesel B100 gasses blanket kinematic viscosity as shown in fig. 10, still in the standard values. It has slightly increased along with biodiesel storage duration. The kinematic viscosity of biodiesel B50 was found still inside of the standard values. It was shown to increase along with the biodiesel storage duration. The effect of the Blanketing system shows that the Biodiesel B100 and B50 CO_2 Blanket was found to have a lower value than the others, while the Natural Air (NA) blanket sample has a higher value than the N2 Blanket. 4. CONCLUSION The results of storing biodiesel B100 and B50 in a blanket with Natural Air, CO2, and N2 gas for 210 days were found to change the characteristics of biodiesel along with the length of storage duration. The addition of CO2 gas blanket was influenced to reduce the oxidation times compared with other gasses blanket, on another side it has increased the value of particle count, TAN, flash point, and density. 5. 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