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Beeswax Material as Corrosion Inhibitor in a Brake Oil System

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Abstract: One of the most important additives used in the braking system is anti-corrosion providing corrosion protection for metals used in braking systems, including cast iron, aluminum, steel, copper, and brass. Beeswax is a biocorrosion inhibitor in braking systems tested. It is cheap, environmental-friendly, a source of nature, and a secondary product in the life of bees. The additive was added in several concentrations of weight percent (wt%), and the method of weight loss was used to study the effect of beeswax as a corrosion inhibitor. The result is an enhancement in corrosion resistance with beeswax content increasing. Many tests, such as an atomic force microscope (AFM), were used to observe the aluminum surface's topography. The result shows a decrease in surface roughness with beeswax additives. Besides, a rheometer and tensiometer were used to study the flow behavior of brake oil/beeswax at different shear rates and shear stresses. It shows a dramatic drop in viscosity as its concentration increases. Beeswax can be considered an environmentally friendly additive and has exhibited a good corrosion inhibitor even in severe acidic environments. The result showed that the brake oil/beeswax fluid offers excellent corrosion protection for metal components and lubricating properties that reduce wear.

Keywords: beeswax, brake oil, anti-corrosion, viscosity, atomic force microscope.

蜂蜡材料作为制动油系统中的缓蚀剂

摘要：制动系统中使用的最重要的添加剂之一是防腐剂，为制动系统中使用的金属提供腐蚀保护，包括铸铁、铝、钢、铜和黄铜。蜂蜡是经过测试的制动系统中的一种生物腐蚀抑制剂。它价格便宜，环保，是大自然的来源，是蜜蜂生活中的副产品。添加剂以几种重量百分比（重量百分比）的浓度添加，采用失重法研究蜂蜡作为缓蚀剂的效果。结果是随着蜂蜡含量的增加，耐腐蚀性增强。许多测试，例如原子力显微镜 (原子力显微镜)，被用来观察铝表面的形貌。结果表明使用蜂蜡添加剂可降低表面粗糙度。此外，还使用流变仪和张力计研究了制动油/蜂蜡在不同剪切速率和剪切应力下的流动行为。随着浓度的增加，它的粘度急剧下降。蜂蜡可以被认为是一种环保添加剂，即使在严重的酸性环境中也表现出良好的缓蚀剂。结果表明，制动油/蜂蜡液可为金属部件提供出色的防腐蚀保护和减少磨损的润滑性能。

关键词：蜂蜡、刹车油、防腐、粘度、原子力显微镜。

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1. Introduction

The brake fluid's main function is to transmit the applied force to the brake pedal, pads, and shoes. The foundation of any hydraulic system is Pascal's Law, which is generally given as "in a fluid at rest in a closed container, a pressure change in one part is transmitted without loss to every portion of the fluid and the walls of the container" [1]. Braihi et al. studied the natural rubber blending with NR/styrene-butadiene rubber (SBR) for petrochemical applications as a surface coating protection [2]. When metal is exposed to oxygen, a thin layer of metal oxide forms on its surface. The reaction of oxygen air forms a dense oxide film. This film "passivates" and protects the rest of the metal by acting as a barrier to reduce further reaction with oxygen significantly" [3]. The formed film partially dissolved by water or other solvents causes a reduction of metal protection. High heat and acids will accelerate the process. Therefore, brake fluid manufacturers also add chemicals to the fluid that stick to and coat the metal surface, providing a barrier in addition to the metal oxide film. The inhibitors are used for corrosion protection for acid solutions to stop the metal dissolution in acid consumption [4]. Several organic compounds with various functional groups have been tested as corrosion inhibitors. Generally, organic compounds significantly affect the level of adsorption on the surface. Therefore, organic inhibitors are used for corrosion inhibitors [5]. Corrosion inhibitors formulated with brake fluid are normally based on an organic inhibitor. The activity of these inhibitors is attributed to their containing polar functional groups, P, S, N, or O [6, 7] in the heterocyclic compounds, and they also may contain π bonding [8]. The polarity function is considered a reaction center for the adsorption process [9]. Molecules containing P, S, N, or O atoms of the inhibitors work in one of two ways [10]: reducing the acid level (neutralizing or buffering amines) and forming a water-repelling barrier film on the metal surface [11]. These corrosion inhibitors are used to protect various metal components of the brake system from the corrosion caused by contact between the metal surface and water absorbed by the brake fluid over time. Many investigations show that the most organic and inorganic inhibitors are toxic, causing hazards to humans and the environment due to their applications [12, 13]. Several researchers focused on replacing these toxic inhibitors with non-toxic compounds. Green corrosion inhibitors protect concrete rebar from corrosion because they are non-toxic, environmentally friendly and available [14, 15]. Green inhibitors are known to be safe, low cost, eco-friendly, and easily available. They can be extracted by simple methods with lower costs than natural products [16]. Beeswax is an organic material of several compounds, mainly oleate palmitate, palmitoleate, and esters, mostly consisting of a long chain of aliphatic alcohols (30–32 carbons) [17, 18]. Regarding its chemistry, it has an approximate formula reported to be $C_{15}H_{31}COOC_{30}H_{61}$. It has a relatively low slipping point in the range of 62-64°C and a density of about 960 kg/m3. Various industrial applications use beeswax, including food protection, cosmetics production, metal injection molding, sealing, and binding [19, 20]. Generally, wax deposition can cause a serious problem through a pipeline system [21], for example, in the distribution of crude oil because, at low temperatures, wax molecules can interact to form a wax aggregate [22]. Bee wax propolis extract showed the inhibition properties on the 304SS in 0.5M sulfuric acid [23], and the inhibitor could retard the corrosion rate of 304SS, which reached 97.29% at 2000 ppm [24]. The effects of wear and corrosion of cast AA6061 aluminum alloy with and without brake fluid were pointed out [25, 26]. The results show that the combined actions of wear and corrosion contribute to the total loss of piston material immersed in brake oil [27]. This work aims to study the effect of beeswax as an anti-corrosion additive for breaking oil systems and a fluidity agent to enhance the flow properties. The beeswax will additive at 0, 0.05, 0.1, 0.5, and 1 weight percentage wt% for brake oil. Weight loss methods will use to understand the corrosion resistance, while an atomic force microscope will apply to the surface of the sample to morphology explanation of inhibitor behavior. Also, flow properties will check by rheometer and tensometer to a deep understanding of the inhibitor mechanism, related energy transmission and consumption.

2. Material and Methods

The following materials were used in the current study:

2.1. Beeswax

Beeswax was used after the extraction process by alcohol to obtain high pure beeswax. In the first, 10 grams of net beeswax were dissolved in 250 milliliters of an alcohol solvent, and the solution was left for 10 minutes to dissolve all the contents of beeswax fully. Then, water was added to the mixture to precipitate the beeswax in the bottom of the solution while other components stayed in the stable mixed state, such as sugar. Therefore, the solution was removed to obtain the precipitate beeswax. Then, repeated this process three times to reach the high purity beeswax. The chemical structure of triacontantyl palmitate $C_{46}H_{92}O_2$ as the main component of beeswax is shown in Fig. 1.

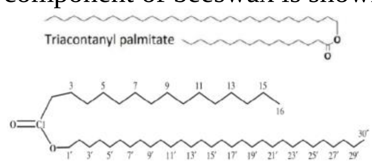

Fig. 1 Molecular structure of triacontantyl palmitate as the major component of beeswax

2.2. Brake Oil

The Venus Lubricants brake oil DOT3 supplied by Canter Lube Oil LLC Company in the United Arab Emirates is shown in Fig. 2, together with the physical properties following the standards of the Department of Transportation in the United States and the J1703 standards of the Society of Automotive Engineers (SAE) for DOT3 type. DOT3 brake oil is glycol-based and does not have borate esters.

2.3. Sample Preparation

Solutions of brake oil/beeswax mixtures have been prepared at 0, 0.05, 0.1, 0.5, and 1 weight percent (wt%) of beeswax on a hot plate and stirrer up to around 60°C for 15 minutes. Then, aluminum plates with 3 cm × 3 cm × 0.6 mm dimensions were cut and cleaned with an alcohol solvent. After the solvent dried, the aluminum samples were weighed by a particular weight device. The test materials were immersed inside solution mixtures and left for 60 days at room temperature. Aluminum plate samples were dried and weighed to measure weight loss to identify the inhibitor's effectiveness.

Fig. 2 The Venus Lubricants brake oil DOT3 supplied by Canter Lube Oil LLC Company in the United Arab Emirates

2.4. Weight Loss Method

In the current study, weight loss measurements were performed on the aluminum samples with a rectangular form of size 3 cm × 3 cm × 0.6 cm in a brake oil sample prepared without and with various concentrations of beeswax. Every sample was weighed by an electronic balance and placed in brake oil/beeswax solution (100 mL). The duration of the immersion was 60 days at room temperature. After immersion, the surface of the specimen was cleaned with double distilled water followed by rinsing with alcohol, and the sample was weighed again to calculate specific weight loss per area.

2.5. Atomic Force Microscope

Atomic Force Microscopy (AFM) Version AA3000 was used to examine the aluminum samples' surface topography, roughness, and the effectiveness of beeswax as an inhibitor for brake oil systems. A small piece of aluminum materials samples was exposed at different brake oil/beeswax mixture ratios, 0.5, 1, 1.5, and 2 wt% of beeswax. The scan size of all the AFM images is 21 μm x 21 μm area at a scan rate of 5 lines per second.

2.6. Cone on Plate Rheometer

The fluidity behavior of brake oil/beeswax solution was studied using the "BROOKFIELD" cone on plate rheometer measurements. The simple cone on plate device provides a uniform rate of the shear rate and a good tool to study viscosity, shear rate, and shear stress for oils. Additionally, it is the most popular instrument for measuring non-Newtonian fluid properties. The cone model number was 40. Therefore, the sample volume was 2 milliliters.

2.7. Tensometer

The surface tension of brake oil/beeswax solution was tested using the JZYW-200B Automatic Interface Tensiometer supply by "BEING UNITED TEST CO., LTD." Direct methods measure the force required to pull, for instance, a metal ring out of a liquid at 25°C.

2.8. Density Device

The density results were obtained using "Matsuhaku." the liquid density mode of the GP-120s instrument. The temperature selected for determining the specific gravity was 25°C.

3. Results and Discussion

3.1. Weight Loss Measurements

Weight loss measurements of aluminum samples against beeswax concentration in brake oil solution were sitting in Fig. 3. The corrosion rate decreases with beeswax loading in a brake oil solution concerning the pure sample. There is a significant drop in corrosion rate with the addition of beeswax until 0.5 weight, while a stable state was shown between 0.5-1 wt%. The diminution in corrosion rate may be attributed to beeswax film adsorption on aluminum plate walls which plays an important role in the insulation of metal against the electrochemical reactions of corrosion. The groups of hydroxyls in beeswax play as heads and attach to the aluminum surface due to polar activity, while a chain of beeswax represents a tie. In other words, the ester group's high polar activity forces of attraction to aluminum surfaces charged beeswax chains to alignment at a vertical angle to an aluminum plate. Increasing beeswax in brake oil solution promotes other beeswax chains' alignment to fill the aluminum surface exposed to brake oil until a maximum percentage around 0.5 wt%, leading to the best corrosion protection result. However, more concentration of beeswax was added to the brake oil solution, but there was no effect on corrosion rate in the semi-stable state with 0.5-1 wt%. That is maybe because all surfaces on aluminum plate surfaces were covered with beeswax chains to an optimal content of about 0.5 wt%. As a result, the best and optimal ratio of beeswax in the brake oil solution was around 0.5 wt%.

3.2. Brake Oil/Beeswax Density Results

The experimental data for the density of beeswax/brake oil solutions at different concentrations of beeswax are illustrated in Fig. 4. The density results were increased with beeswax loading in brake oil solution for all samples but the fluctuation method. The decreasing density behavior may be linked to high immiscibility between beeswax chains and brake oil molecules at room temperature. Therefore, these phenomena encourage isolation and deposition for high molecular weight beeswax chains to produce a high solution density. The unstable readings of beeswax/brake oil solution density can be attributed to the high range of molecular weight of beeswax chains caused to a different level of beeswax molecules agglomerations in solution. However, the aging of acidic solution with aluminum metal also results in corrosive products.

Additionally, Fig. 3 shows the higher result of the density of beeswax percentage around 0.1 wt%, which introduces sufficient sizes of beeswax chains to the insulated aluminum wall, while the lowest value was reached in content around 0.5 wt%, which appeared to be the best protection barrier. Accordingly, there is an interesting relation between weight loss and density measurements, especially at the proposed optimal concentration of about 0.5 wt%.

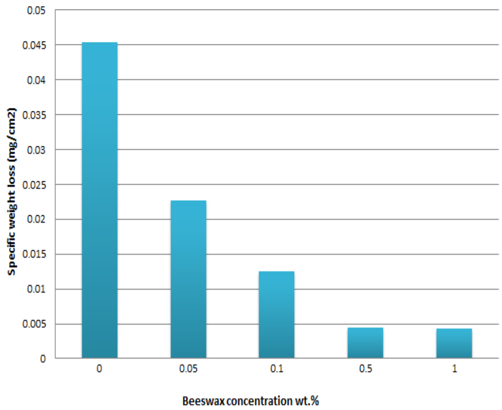

Fig. 3 Weight loss of aluminum plate samples after the immersion in brake oil with/without beeswax at different concentrations for 60 days

3.3. Characterization by Atomic Force Microscope

Fig. 5 represents three- and two-dimensional images of aluminum plate surfaces from an atomic force microscope after and before immersion in beeswax/brake oil solution at different concentrations (0, 0.05, 0.1, 0.5, and 1 wt%) of beeswax. These figures provide evidence about the anti-corrosion performance of beeswax in case of the shape of corrosion attack morphology, distribution, and depth on the aluminum surfaces. They showed a significant decrease in size, amount, and depth of corrosion carvings with the increasing beeswax content in the brake oil solution. Firstly, in Fig. 5A and 5G, the aluminum plate's smooth surface before immersion in a brake oil solution represents the normal morphology of aluminum. However, the corrosion attack appears clearly in Fig. 5B and 5H, which assimilate aluminum plate inside brake oil solution without any additives. So, the pure brake oil showed the highest degree in number, depth, and distribution of corrosion effects in all-aluminum samples. Using beeswax as an additive in brake oil solution changed the surface morphology toward the low value of number, shape, and distribution of corrosion products on the sample's plates.

This effect of beeswax results from the high adsorption ability of aluminum surfaces due to their containing ester groups.

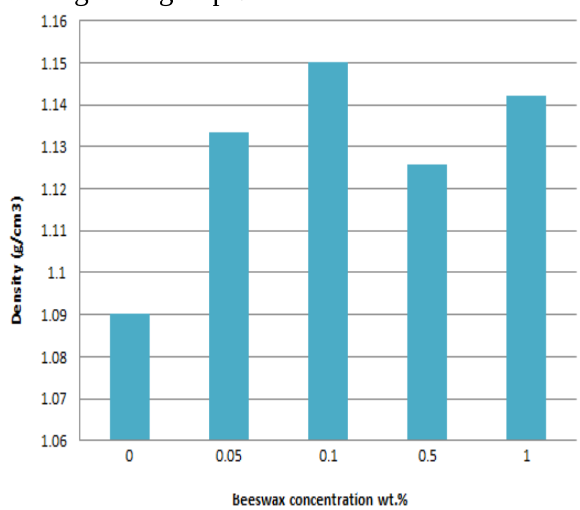

Fig. 4 Density measurements of brake oil with/without beeswax at different concentrations after the immersion of aluminum plate samples in the solution for 60 days

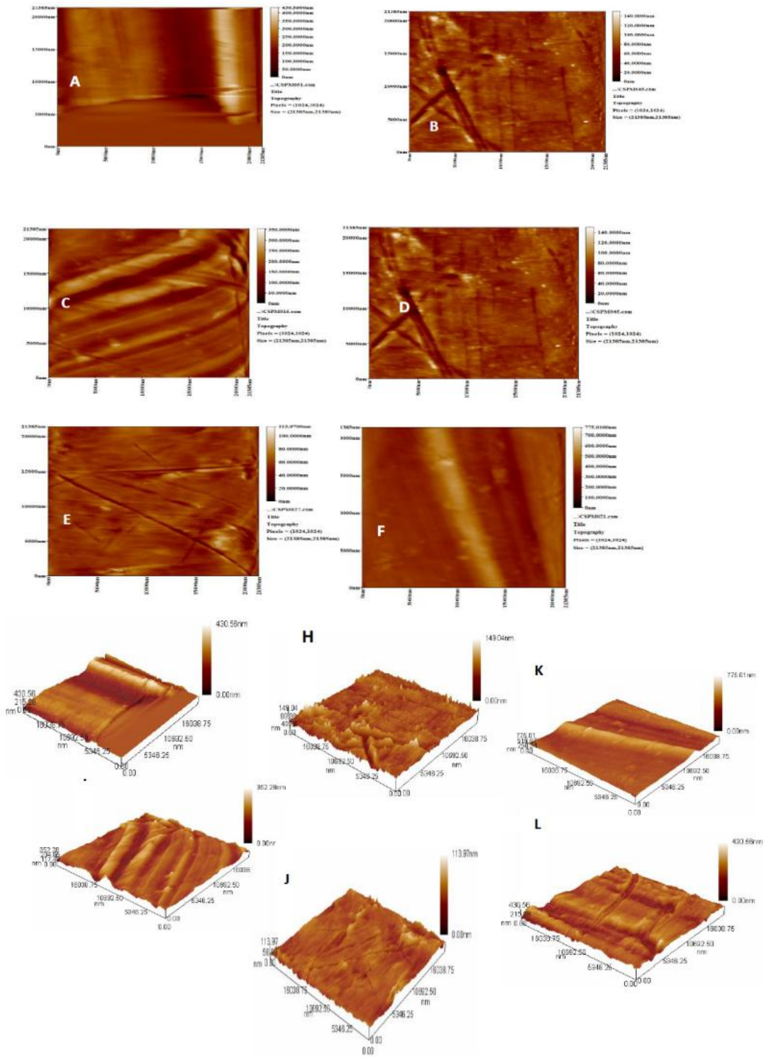

Fig. 5 Two- and three-dimensional images of the samples from atomic force microscope after the immersion in beeswax/brake oil solution at different concentrations of beeswax for 60 days: before immersion - A and G = 0 wt%, after immersion - B and H = 0, C and I = 0.05, D and J = 0.1, E and K = 0.5, F and L= 1 wt%

These function groups have a high affinity to adsorb on the aluminum wall so that the beeswax plays an efficient barrier between brake oil liquid and aluminum surfaces. In low beeswax concentration, the corrosion effect becomes less and little in lines shapes of corrosion attack, which may be attributed to insufficient amount of beeswax to cover all aluminum surfaces, for example, in 0.05 wt% in Fig. 5B and 5H. Therefore, using more concentration makes their corrosion product lines reach a lower value, as in a percentage of 0.1 wt% in Fig. 5C and 5I, because the additive coated more spaces on aluminum samples. Moreover, the more decreasing corrosion effect morphology was found at a percentage of 0.5 wt%, and this result fits the weight loss and density measurements, as shown in Fig. 5D and 5J. On the other hand, the corrosion products began to appear in the highest beeswax content, as shown in Fig. 5F and 5L. This increase can be explained according to high-level agglomerations of beeswax chains inside brake oil solution due to the high molecular weight and content in the solution. Therefore, the ability of additives to adsorbs on aluminum surfaces decreases to encourage corrosion attack of brake oil on aluminum samples.

3.4. Viscosity, Shear Rate, and Shear Stress Results

Viscosity behavior of brake oil/beeswax mixtures concerning the concentration increasing of beeswax have been represented in Fig. 6. The shape showed a dramatic decrease in solution viscosity of brake oil with increasing beeswax content. The decline manner of solution viscosity can be explained due to the adsorption and formation of beeswax film on aluminum walls, resulting in an excellent barrier layer against brake oil solution. In more detail, the high ability of chain alignment in the vertical direction to the aluminum wall permits lubricant efficiency with applied force. Additionally, this film plays an important role in friction loss as a lubricant factor and the related energy consumption in the brake system. So, beeswax may be an important anti-corrosion additive, especially in dynamic corrosion conditions, because it has an interesting effect on the friction forces of breaking oil with the aluminum wall.

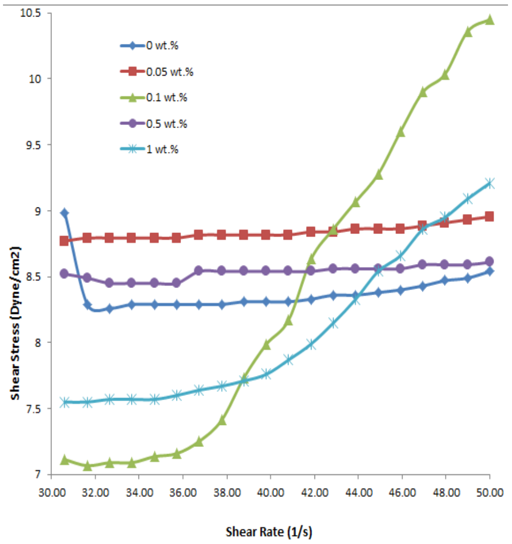

Fig. 6 Viscosity behavior of brake oil with/without beeswax at different concentrations after the immersion of aluminum plate samples in it for 60 days

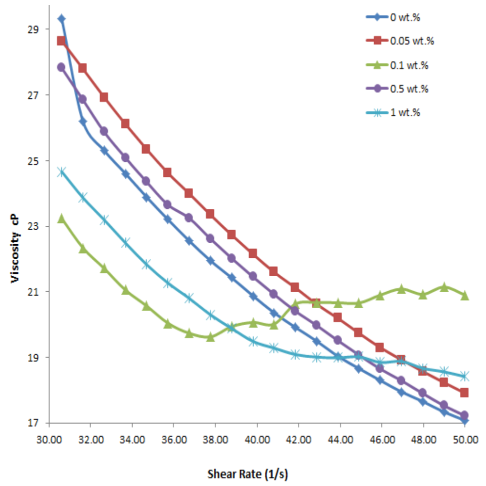

Fig. 7 Shear stress and shear rate relationship of brake oil with/without beeswax at different concentrations after immersing aluminum plate samples in it for 60 days

The relationship between shear rate and shear stress of pure brake oil and brake oil/beeswax solutions with different ratios of beeswax is reported in Fig. 7. Two stages of flow behavior are illustrated: stable state or Newtonian flow properties and shear theming behavior. Pure brake oil sample and lower concentrations of beeswax in solution behave similarly to Newtonian flow characteristics. However, there are exceptions, especially in the beginning and end of pure samples, which may be attributed to technical issues of the flow tests. On the other hand, the shear-thinning flow was found in the high loading of beeswax in solution at 0.5-1 wt% due to the high effect of beeswax chain arrangements in brake oil solution. Also, there are some exceptions, especially at the start of measurements, which appear as a stable state or Newtonian flow. This manner can belong to a low shear rate which does not permit the molecules to take their new positions.

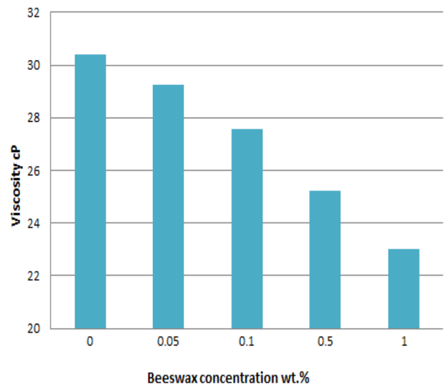

Fig. 8 Viscosity behavior at increasing shear rate of brake oil with/without beeswax at different concentrations after immersing aluminum plate samples in it for 60 days

The viscosity manner with shear rate changing of pure brake oil and brake oil/beeswax solutions at different beeswax content was illustrated in Fig. 8. Generally, the figure shows a decrease in brake oil solution viscosity with an increasing shear rate for all sample percentages except 0.1 wt%, which is fluctuated behavior. Decreasing viscosity with shear rate and shear-thinning behavior may occur due to disengagements and sliding of beeswax chains with brake oil molecules in the solution. Firstly, the lower sample content, for example, 0.05-0.1 wt%, has more viscosity than the pure sample because the beeswax percentage is not sufficient to affect flow properties. Secondly, the higher concentration of beeswax in a brake oil solution, for example, 0.5-1 wt%, turns the flow behavior of solution viscosity depending on shear rate to a lower viscosity level. This behavior may be attributed to sufficient ratios that fill all spaces inside brake oil liquid. However, the fluctuation situation of 0.5 wt% indicates a better condition for using this optimal concentration at a lower shear rate around (30-38) s^{-1}.

3.5. Surface Tension Measurements

Surface tension data of pure brake oil and beeswax/brake oil solutions at different concentrations of beeswax was illustrated in Fig. 9. Generally, there are decreases in surface tension measurements with loading beeswax in the solution compared to the pure sample, except for the last percentage, which had a high surface tension value. The decline behavior may be attributed to high adhesive forces between groups of beeswax chains and brake oil molecules, producing additive chain dispersion in solution. In low additive content, there is more chance of beeswax chains for dispersion in solution and becoming individual molecules resulting in higher adhesive forces than cohesive forces between brake oil molecules. Therefore, the most interesting decrease in surface tension readings is at a percentage of 0.05 wt% while becoming a slight degree in decline with another ratio such as 0.1 wt%. Moreover, using the other beeswax concentration, for example, 0.1-0.5 wt%, turns the behavior toward the surface tension increasing in comparison with the 0.05 wt% sample. The going up of behavior can be attributed to beeswax chains agglomeration due to full spaces inside brake oil solution by additive molecules. Also, film formation was made by these chains agglomerations, and the cohesive forces reached a higher value. In addition, the shape showed the highest surface tension value at 1-wt% beeswax content due to beeswax overload in the brake oil solution and more agglomeration and sedimentation of chains in the solution mixture. In other words, the best content is recommended at about 0.1 wt% because this concentration has the greatest decrease in weight loss, and the surface tension value remains under a pure sample.

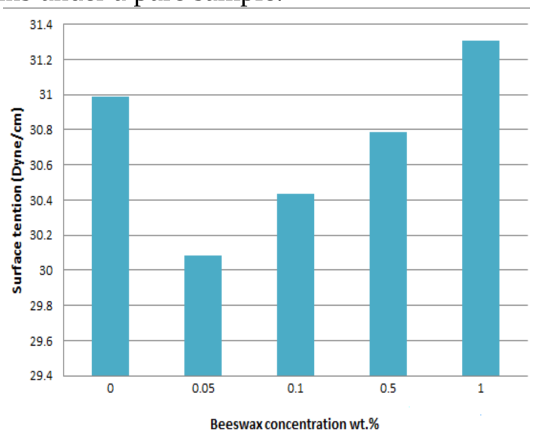

Fig. 9 The surface tension of brake oil with/without beeswax at different concentrations after the immersion of aluminum plate samples in it for 60 days

4. Conclusions

The corrosion in the braking system is one of the most important issues. Brake fluids must, for example, not corrode or chemically react with any materials in the hydraulic system. This work revealed that the acidity of brake oil decreases with the addition of beeswax due to the interaction of acidic hydrogen with the ester group in beeswax without effect its ability as brake oil. Beeswax shows good miscibility with the brake oil due to the polar interaction between both of them. The low specific heat of beeswax (Beeswax = 3.4 J/g. °C, Aluminium = 0.436 J/g. °C) transfers it from solid to liquid states and vice versa, even with low heat requirements; thus, it is easy to cover the metallic part of the brake oil system. It has lubricant properties, thus facilitating the movement of the oil, and good ability to form a very thin film on the material surface to inhibit corrosion and erosion.

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