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#### Review Article

## The Grease Formulation Using Waste Substances from Palm Oil Refinery and Other Industrial Wastes: A Review

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### ABSTRACT

Many applications use Spent Bleaching Earth (SBE) despite being considered hazardous waste from the palm oil refinery process. Its production increases yearly, similar to waste cooking oil (WCO). The SBE is known as a thickener in grease formulation. The same goes for red gypsum, waste motor oil, stearic acid, and lithium hydroxide monohydrate. They are all considered thickeners but have different durability in protecting base oil in grease. Then, previous studies revealed their performances with side effects detection against the environment and human bodies. Cooking oil is a heat transfer medium for serving foods with higher amounts of unsaturated fatty acids. The number of fatty acids might change after cooking oil consumption and become highly demanded due to the chemical properties of density, viscosity and fatty acids. Nowadays, people lack awareness

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ISSN: 0128-7680 e-ISSN: 2231-8526 of the importance of recycling palm oil waste. They intend to dispose of it instead of recycling it for sustainable energy resources. Therefore, this paper will discuss the grease formulation, contaminant available in WCO, its treatment, issues regarding different thickener consumption, treatment against Spent Bleaching Earth (SBE), and propose the safe thickener and additives for future intakes. This study found that adding Fume Silica (F.S.) as a thickener and Molybdenum Disulfide (MoS<sub>2</sub>) enhanced the grease stability. Further treatment against SBE (remove residue oil) and WCO (metal elements, undesired impurities and water content) is necessary for providing good quality formulated grease.

Keywords: Grease formulation, spent bleaching earth, waste cooking oil

#### **INTRODUCTION**

The occurrence of environmental pollution over the years has become a big concern among scholars and researchers. The ongoing generation of waste products, whether from domestic or industrial sources, has become a significant problem. Therefore, this subject has sparked a debate among them. The waste management process is seen as expensive, and several manufactured pollutants contribute to environmental damage. Without effective management, pollution may occur and cause an ecological system imbalance. According to Lopes et al. (2020), the generation of waste cooking oil (WCO) in highly populated countries such as Japan (0.57 million tonnes), India (1.1 million tonnes), the United States of America (1.2 million tonnes), and China (5.6 million tonnes) has increased considerably. According to estimates, Malaysians squander 540 000 tonnes of WCO composed of vegetable and animal fats yearly without treating them as waste (Suzihaque et al., 2022). Meanwhile, although most households know the importance of recycling WCO, only a tiny percentage recycle it. In 2020, the Domestic Trade, Cooperatives, and Consumerism Ministry (KPDNKK) stipulated that around 45 million kg of cooking oil was utilised in a month, which means around 1.45 kg of cooking oil was consumed by each person per month (Farid et al., 2020). Meanwhile, another study found that the average monthly WCO production per family is estimated at around 2.34 kg, according to government statistics (Nizam & Misdan, 2022). Figure 1 shows the Waste Cooking Oil Production (million tonnes) within different Countries in 2019.

Pollution can have negative environmental impacts by causing eutrophication, blockage of a municipal drainage system, problems with water treatment facilities, and degradation in aquatic animals due to lack of oxygen supplied by a surface blockage by waste cooking oil (Singh-Ackbarali et al., 2017). Water contamination occurs when WCO is disposed of into the sewer system, which creates obstructions that prevent wastewater from flowing to treatment facilities. If WCO



*Figure 1*. Bar chart for the waste cooking oil production of different countries (Lopes et al., 2020)

is disposed of in municipal solid waste landfills, it will pollute the soil (Feo et al., 2020). For example, the affected operator spends \$3.5 million yearly to clear municipal pipes of blockage caused by waste oil pollution (Vollaard, 2017). It has an impact not only on the environment but also on the cost of treatment.

Recently, many cases have been reported about improper disposal of waste cooking oil. People's lack of awareness contributes to this disposal problem. A survey of respondents in the University of Malaya Cafeteria revealed that approximately 50% threw WCO into the sink, 35% threw it as normal waste, and 20% knew of the impact of repeated consumption (Noor & Hua, 2016). The fluctuating price of mineral oil has sparked debate among academics. However, a non-linear relationship between oil prices and market deliveries due to insufficient production and spare refinery capacity was found to reduce crude oil volumes over the long term. (Dees et al., 2008). Many researchers have found the tendency of WCO as raw material in tuning new products via chemical processing, such as biodegradable polyurethane sheets, greases, bio-lubricants, soaps and alkyd resins, to address these issues (Panadare & Rathod, 2015).

Waste cooking oil (WCO) is defined as used cooking oil after the cooking process. It is generally made of edible oil containing 95% triglycerides, glycerol and fatty acids linked to hydroxyl groups via ester linkage (Vitz et al., 2021). It is categorised as lipids and results from the esterification process. Anderson (1991) states that WCO as a lubricant has been applied since ancient times. This oil acts as a lubricant during operation, reducing the frictional force between two surfaces (wheels and gears). During the Pharaonic period, around 1400 B.C., Hittite chariots used the WCO on the axle (Sharma & Singh, 2019). This application was widely used until 4000 years ago, but it was discontinued in the 19th century due to the higher chemical properties conceived by mineral oil.

Even though mineral oil outperforms waste oil in terms of chemical properties and performance, researchers still have room to find a better way to use waste products as sustainable energy. To maintain grease quality, having a good product free of unwanted impurities should be a priority in grease formulation. It impacts the appearance, colour and performance of grease produced. Furthermore, most waste oils contain undesirable impurities compared to treated crude oil. Many WCOs are taken from various resources until they can impact the tradition and method of cooking styles within various races. Furthermore, the frying period, temperature, and material used in cooking can all affect the properties and type of impurities present. It can be difficult because the process necessitates additional treatment against WCO. WCO, in particular, must be treated before being used as a base oil.

Spent Bleaching Earth (SBE) is used as a thickener known as palm oil waste. WCO is classified as palm oil waste and is used as a base oil in grease formulation. Furthermore, they can reduce environmental pollution by recycling. Not only that but red gypsum,

waste motor oil, stearic acid, and lithium hydroxide monohydrate are also considered thickeners derived from industrial waste. Unfortunately, some of them could not perform well as thickeners, and several adverse effects are found when using these substances for the grease formulation. Then, the SBE is found to be highly hazardous due to several toxicant impurities (Fattah et al., 2014). Further treatment needs to be done to remove these impurities to make it safer.

#### **Contaminant Available in WCO**

To strengthen the issues, Mannu et al. (2020) stipulated some impurities in used cooking oil (WCO), e.g., metal traces, organic molecules and spices after being heated or used. The physical properties of cooking oil also changed after use, especially in terms of colour and viscosity (Jaarin & Kamisah, 2012). Therefore, oxidation products from used oils become unfit for human consumption (Tan et al., 2011). Several investigations on fatty acids have been conducted to assess the stability of fatty acids and the efficacy of vegetable oil as a lubricant.

According to Godson and Vinoth (2015), the two most common saturated fatty acids in WCO are palmitic acid and stearic acid. Meanwhile, myristic acid, lenolenic acid, capric acid, and lauric acid are found in small amounts (Figure 2). Furthermore, not only are metal traces and impurities present, but the generation of fatty acids is also classified. According to Awogbemi et al. (2019), the number of fatty acids will degrade before and after WCO consumption. The study discovered that the percentage of oleic acid in sun foil, palm oil, and sunflower oil was the lowest but the highest in margarine. Oleic acid is known as an oxidation resistor (Hernandez, 2016). The acid may stop oxidation. Therefore, it is commonly found as the highest level per cent in WCO after consumption as it maintains the oil presence.

The theory regarding the metal element production from heating cooking oil is acceptable since many studies have traced the availability of these elements using Graphite Furnace Atomic Absorption Spectrophotometry (GFAAS). GFAAS can trace metal elements inside edible oils or foods and detect arsenic in natural medicine products (Khalid et al., 2016). Trace elements concentration of edible oils might be lower in quantity, e.g., arsenic (As), Cadmium (Cd), Lead (Pb), Selenium (Se) and Chromium (Cr). They are toxic



*Figure 2*. Pie chart of the common components in WCO (Godson & Vinoth, 2015)

and have a negative impact on consumer health (Manarattanasuwan, 2011). Copper and iron also can be detected in edible oil using (GFAAS) (De Leonardis et al., 2000). The saponification method is similar to the atomic absorption spectrophotometer (AAS) (Handajani et al., 2014).

#### **Treatment on WCO**

The treatment of WCO is crucial in removing severe undesired impurities present in it. Some researchers found the best way to remove the undesired impurities. The oil will face degradation in quality due to continuous consumption. The longer the continuous repetition of oil consumption, the higher the amount of saturated fatty acid in that oil, which may protect from oxidation easily (Hernandez, 2016). A study by Rajvanshi and Pandey (2016) purified WCO via a filtration method using filter paper. The filter paper is put into a funnel to separate severe undesired impurities. Then products from the filtering process (filtered oil) need to be appropriately kept (sealed container) to avoid a reaction from the air. Since the filtered oil is being filtered, it has similar properties to net cooking oil (Rinaldi et al., 2017). Additional treatment is required by heating the WCO within an hour to reduce moisture content. The oil must be kept in a sealed container and away from the air to prevent rancidity. Other studies also involved the treatment of WCO into a toilet cleaner product, using a filtration process with three types of absorbents (Kamaruzaman & Zin, 2019). The study also claimed similar properties between fresh and filtered cooking oil, but filtered cooking oil is highly safe to use due to less peroxide value (Kamaruzaman & Zin, 2019).

Peroxide value is the number of oxygen in peroxide, which can be measured in the oxidation process against cooking oil. Generally, it is also defined as hydro-peroxides in substances and can be used to determine the deterioration rate of oil (Gordon, 2004). Therefore, good oil quality, especially for grease formulation, depends on the amount of peroxide value related to the oxidation process, which reveals the good preservation status of oil. Fresh cooking oil (FCO) comprises a higher peroxide value due to the storage process (Almeida et al., 2019). Then, oxidation is a major problem against the selected based oil for grease formulation. The higher peroxide value can be described as being mostly available in fresh oil. First, treating WCO before reusing it to produce good quality formulated grease with minimum degradation effects is important. Awogbemi et al. (2019) stated that there would be a higher interchange of density, fatty acids and viscosity after cooking oil consumption. Using oil with a low peroxide value, "waste oil," is recommended to avoid oxidation.

### **Grease Formulation**

Usually, the grease formulation comprises six major steps, from collecting base oil to selecting the final grease. Grease is formulated using base oil, thickener, and additive. It is important to design these steps to make sure the generated grease product is highly defined

Muhammad Auni Hairunnaja, Mohd Aizudin Abd Aziz, Nur Amira Fatihah Bashari, Mohd Azmir Arifin, Navinash Nedumaran, Khairuddin Md Isa and Umi Fazara Md Ali

based on acceptable industry standards of common grease acceptable properties via the International Organization for Standardization "ISO 12924 and ISO 6743-9". Grease is chosen based on its structure and NLGI number. The highest composition should be base oil (waste cooking oil), thickener, and additive. In order to produce grease, the heating process must be completed within three hours using a homogeniser and a hot plate. The greases produced will be tested using the American Society for Testing and Materials (ASTM) method and SKF's grease test kit before being finalised as selected grease. However, grease performance is determined by the type of thickeners and additives used within adequate composition values. Figure 3 shows the flow chart of the general overview of the grease formulation steps.



Figure 3. Flow chart of the general overview for the grease formulation steps

Pertanika J. Sci. & Technol. 31 (5): 2505 - 2529 (2023)

#### Issues in Using Stearic Acid and Lithium Hydroxide Monohydrate as Thickeners

A grease formulation from WCO had been produced by Othman (2009) in combining stearic acid and lithium hydroxide monohydrate as thickeners for different ratios. The study also involved maintaining WCO and sodium diethyl dithiocarbamate as an antioxidant additive. The study also discovered that increasing the thickener impacted the grease performance significantly in terms of NLGI hardness value, work penetration value, operating temperature, and dropping point value, but not for copper corrosion testing. Therefore, the thickener was directly proportional to these grease performance values. In contrast, the result showed that adding additives would significantly impact the metal corrosion up to 4c grade, which is highly corrosive. Lowering the amount of additive denoted the grease's ability to protect the metal surface from chemical attack.

Unfortunately, lithium hydroxide monohydrate is not safe to use in grease formulation due to its limitation stability under normal storage temperature conditions (Livent, 2018), which means it is vulnerable to igniting or exploding and needs to be kept in a cool and dry place. Even though it can form a high-temperature grease, the New Jersey Department of Health and Senior Service (2014) stated that it is a highly corrosive chemical that can cause irritation and burn the skin and eye, leading to eye damage. A pungent odour can irritate the nose and throat, damage the lung, and shorten the breath.

Stearic acid is an emulsifier, emollient, and lubricant that can help prevent the product from separating. It is known as a thickener and can stabilise the grease structure, which is parallel to thickener properties and helps in smoothing the grease structure (Delaneau, 2021). Even though the combination of stearic acid and lithium hydroxide monohydrate (based) can produce a thickener with fewer acidity properties (neutralisation), which is not corrosive, with high mechanical stability and excellent water resistance, the issue cannot be dismissed due to the difficulty in handling these substances during operations. Users can easily spill a bulk powder of lithium hydroxide monohydrate, causing waste and possible respiratory irritation. Another disadvantage of applying this substance is the possibility of yielding grease composition, which may result in lower than desired specifications throughout the spillage and tends to clump and form if exposed to water or high humidity storage area (Smith et al., 2000). Recently, no studies on the toxicity of the stearic acid substance were mentioned, even though it had been tested against animals (Libert, 1987). However, some studies have suggested that other side effects may occur in a few people, such as irritation of sensitive skin, the possibility of carcinogen agents (causing cancer), or toxin build-up in the brain or organs.

#### Issues in Using Spent Bleaching Earth as a Thickener

A WCO study using Spent Bleaching Earth (SBE) as a thickener found no significant impact on oil (Abdulbari et al., 2011). The waste can be converted into a new product but requires Muhammad Auni Hairunnaja, Mohd Aizudin Abd Aziz, Nur Amira Fatihah Bashari, Mohd Azmir Arifin, Navinash Nedumaran, Khairuddin Md Isa and Umi Fazara Md Ali

specific treatments to remove most potential fire and pollution hazard impurities: 20-40% of residual oil by weight, organic compounds, and metallic impurities (Rokiah et al., 2019). Using this sticky material is brilliant since it can save costs from purchasing industrial thickeners for grease formulation while also minimising the environmental impacts of disposal issues. According to a recent study, the SBE consists of fume silica within the range of 50% to 83.05% (Dermawan & Ashari, 2018), making it an excellent thickening agent (Barthel et al., 2005) with no toxicity impact (fume silica) and as anticaking agent (Younes et al., 2018). The study discovered that while SBE can hold base oil at room temperature, it cannot hold WCO due to temperature and pressure changes. So, even though it is a fine particle of clay capable of absorbing and retaining oil, these are the criteria required by many thickeners, and this is due to the oxidative properties of WCO. Therefore, according to the final study assumption, some additives should be added to prolong this issue. SBE is also classified as solid hazardous waste because of its high organic content, which exceeds the waste acceptance criteria (WAC) for hazardous waste landfills by 6% (Fattah et al., 2014). Another issue with this substance is the difficulty in disposing of it due to the high amounts of the water-insoluble substance, diverse compositions, macro-elements, and micro-elements associated with SBE (Piotrowska-Cyplik et al., 2013). A study on mice confirmed the corrosive effect of SBE application on the skin, and the results showed its tendency to improve skin lesions (Yang et al., 2017). Even though it is classified as highly hazardous waste, the Indonesian government has declared it is only classified as nonhazardous waste if it contains less than 3% oil (Jong, 2021). Nonetheless, the study did not initially treat SBE other than by removing stone and agglomerate particles before the grease formulation. As a result, the grease produced by the SBE substance can be extremely hazardous due to its toxicity and may cause skin irritation when touched. Table 1 shows a Summarisation of using SBE as a thickener.

Table 1

Full Name	Spent Bleaching Earth (SBE)
Problem Against SBE	• Most potential fire and pollution hazard impurities comprise 20-40% of residual oil by weight, organic compounds, and metallic impurities.
Characteristic of SBE	<ul> <li>Sticky substances.</li> <li>Comprises fume silica within the range of 50% to 83.05%.</li> <li>Anticaking agent.</li> <li>SBE can hold base oil at room temperature; it cannot hold WCO due to temperature and pressure changes.</li> </ul>
Other issues related to SBE	<ul> <li>SBE is also classified as solid hazardous waste because of its high organic content, which exceeds the waste acceptance criteria (WAC) for hazardous waste landfills by 6%.</li> <li>Another issue with this substance is the difficulty in disposing of it due to the high amounts of the water-insoluble substance, diverse compositions, macro-elements, and micro-elements.</li> </ul>

Summarisation on using SBE as a thickener

Grease Formulation from Palm Oil and Other Industrial Wastes

Table 1 (continue)	
Full Name	Spent Bleaching Earth (SBE)
Research Relating to SBE	• SBE can cause a corrosion effect on the skin and may ameliorate the skin lesion.
Another opinion about hazardous SBE.	• The Indonesian government has declared it is only classified as non- hazardous waste if it contains less than 3% oil.
The problem with this study	• The study did not initially treat SBE other than by removing stone and agglomerate particles before the grease formulation.

#### Issues in Using Red Gypsum/Fumed Silica (F.S.) as a Thickener

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Another study of grease formation with waste oil and red gypsum/fumed silica (F.S.) within different ratios by Razali et al. (2017) has revealed the red gypsum's ability to hold different waste oils. The waste oil involved is WCO, used oil, silicon oil, and waste emulsions. After two months of storage, an oil separation occurred even with a ratio of 50% (red gypsum)/50% F.S. of WCO grease with 50 ml oil separation. In contrast, waste emulsion oil with 10 ml oil separation revealed the best result of grease within a similar ratio after the same period. As a result of its high oxidation properties, red gypsum cannot hold enough WCO structure.

#### Issues in Using Waste Motor Sludge as a Thickener

The study on grease from WCO and Waste Motor Sludge by Rajvanshi and Pandey (2016) proved the compatibility of WCO as a base oil for the grease production process. The study was also tested using different mixing ratios between lithium and motor sludge within various amounts of WCO and including a few quantities of molybdenum disulfide (MoS<sub>2</sub>) as an additive. Hence, the results showed that the mixing quantity of these substances significantly impacted the increment of the dropping point and work penetration value by having directly proportional values. The grease samples also revealed a minor corrosive nature, especially under the specific static condition when tested by the ASTM method within the slightly tarnished appearance of stage 1B. Still, the dropping point decreased significantly when the amount of lithium and motor sludge was reduced while the WCO was increased. In contrast, lithium and motor sludge have been detected when lithium is very unstable and needs to be mixed with the most stabilising agent, such as "motor sludge" (Sani & Florillo, 2020). Commonly, it needs to be kept within mineral oil as it easily reacts to moisture in the air (Dye, 2021). Motor sludge can resist high temperatures due to its properties which can withstand heat. At the same time, it is highly temperature resistant as it can cause damage to the car engine when it is not frequently removed. It can be a stabilising agent in storing lithium within the sticky characteristic. Furthermore, lithium also has superior heat-transfer fluid properties, especially for high-power nuclear reactor density (Dye, 2021) with higher specific heat capacity (Szelong & Fan, 2020).

Unfortunately, lithium is still highly unstable, with a high explosion risk if not handled gently during or after grease formation. Inappropriate substance portions cause the grease to explode during operation.

The motor sludge is considered highly hazardous due to several chemical elements present in its compositions like heavy metals group, including nitrogen, chromium, Phosphorous, Potassium, Iron, Copper, Calcium, Magnesium, Cadmium, Phosphate, Chromium, barium, lead, zinc, mercury, chromium, arsenic, Zinc and Sodium (Johnson & Affam, 2019). The treatment of motor sludge can be done, but it takes a very long process to completely treat a large area (Johnson & Affam, 2019). Furthermore, the research used untreated motor sludge, a significant hazardous waste (waste lubricating oil can cause skin cancer) and lithium. This unstable chemical substance is unpredictable due to its explosive ability for the upcoming grease formulation process. Table 2 shows a summarisation of using Waste Motor Sludge as a thickener.

8	5
Full Name	Waste Motor Sludge
Characteristics of Waste Motor Sludge	<ul> <li>Motor sludge can resist high temperatures due to its properties which can withstand heat.</li> <li>It can cause damage to the car engine when it is not frequently removed.</li> <li>Motor sludge can be a stabilising agent in storing lithium within the sticky characteristic.</li> </ul>
Hazardous Waste Motor Sludge	• It is considered highly hazardous due to several chemical elements in its compositions, like the heavy metals group.
Characteristics of Lithium	• Has superior heat-transfer fluid properties, especially for high-power nuclear reactor density with higher specific heat capacity.
Hazardous of Lithium	<ul> <li>Considered a highly unstable material, with a high explosion risk if not handled gently during or after grease formation.</li> <li>Inappropriate substance portions cause the grease to explode during operation.</li> </ul>
Other opinions about Waste Motor Sludge	• The treatment of motor sludge can be done, but it takes a very long process to treat within a large area completely.
The problem with this study	<ul> <li>This research used untreated motor sludge, a significant hazardous waste (waste lubricating oil can cause skin cancer).</li> <li>Lithium is an unstable chemical substance, which is unpredictable due to its explosive ability for the uncoming grease formulation process.</li> </ul>

Table 2

Summarisation on using	Waste Moto	r Sludge as a	thickener
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#### Summarisation of Comparative Studies Between All the Mentioned Research

Table 3 summarises comparative studies of four studies in grease formulation using WCO.

#### **Future Prospect**

Recycling WCO as a base oil for grease production will encounter pollution problems due to improper waste disposal. Furthermore, using WCO to make grease is more cost-

Table 3 Summarisation of comparat	ive studies of grease formulation			
Parameters involved in grease analysis / Previous Study of WCO grease	Norasimah Mohd Othman (2009)	Abdulbari et al. (2011)	Razali et al. (2017)	Rajvanshi & Pandey (2016)
Title	Production of grease from waste cooking oil	Lubricating grease from spent bleaching earth and waste cooking oil	Synthesis of grease from waste oils and red gypsum	Lubricating grease from waste cooking oil and waste motor sludge
Thickener and Additives	Stearic acid and lithium hydroxide monohydrate (thickener) diethyl dithiocarbamate (additives)	F.S. & SBE (Thickener)	red gypsum (thickener)	lithium & motor sludge (thickener). mos <sub>2</sub> (additives).
Consistency test (NLGI Grade) Cone Penetration Test	The study found that adding only LiOH may increase the NLGI value of grease. Meanwhile, having the increment combination ratios between LiOH and stearic acid may increase the work penetration test value, which defines the lower grease consistency value.	The study found that adding WCO and SBE may significantly result in up to 3 NLGI values. Nevertheless, the NLGI result will increase up to 4 when adding F.S. In order to get around 2–3 NLGI values, the SBE composition should be the major part, followed by WCO and F.S.	NGLI value is 1, which is not for the sample WCO grease	the study involved formulating grease at different mixing times. therefore, this led to the consistency of nlgi value at 1, but the consistency value dropped suddenly to 0 at the mixing time of 5 hrs.
Oil Bleeding	N/A	N/A	N/A	N/A
Oil Separation	N/A	N/A	50 ml oil still separated from grease within thickener of 50/50 fumed silica: red gypsum presence.	N/A
Dropping Point	Increasing the amount of LiOH might elevate the dropping point of the grease.	The formulated grease produced has no dropping point, meaning the grease resistance exceeds 350°C.	Applied but not on sample grease of WCO.	The formulated grease had high-temperature resistance above 200°C even at different mixing times.

Grease Formulation from Palm Oil and Other Industrial Wastes

Pertanika J. Sci. & Technol. 31 (5): 2505 - 2529 (2023)

Rajvanshi & Pandey (2016)	All the grease samples appeared slightly tarnished, under 1B of the ASTM rating.	N/A	N/A	Used to be applied at general machines without extreme pressure.	This study focuses on generating a new grease product from waste substances to minimise the burden of utilising conventional sources.
Razali et al. (2017)	N/A	N/A	N/A	Sealing of bearing against loss of lubricant acts as a sealant to minimise leakage.	The study was to develop grease from different waste oil. Unfortunately, WCO is not compatible with red gypsum.
Abdulbari et al. (2011)	The corrosive test for grease within or without F.S. revealed that all the copper strips fell in class 1b (slightly tarnished).	Grease without F.S. possesses a temperature resistance of up to 200°C, while grease within F.S. developed a temperature resistance of up to 150 °C or more.	The formulated grease without F.S. resulted in a low frictional coefficient value (0.095) compared to the grease plus F.S. resulted in a value of (0.11). A lower frictional coefficient value is highly desirable.	Medical facilities, aircraft and other heavy industries applications.	It is multipurpose grease designed to fit in extreme operating conditions such as high temperature or cold temperature climate and extreme pressure.
Norasimah Mohd Othman (2009)	Having a huge number of additives can highly affect the corrosiveness of copper strips within grade 4c.	N/A	N/A	Bio-based grease for agriculture/farming, forestry and food processing.	The study found that adding LiOH may increase the NGLI and dropping point value, but adding the Stearic acid may help to get the desired grease hardness.
Parameters involved in grease analysis / Previous Study of WCO grease	Copper Corrosion	Thermal stability Analysis Using Thermogravimetric Analyzer (TGA)	Tribology Properties	Application	Explanation

Muhammad Auni Hairunnaja, Mohd Aizudin Abd Aziz, Nur Amira Fatihah Bashari, Mohd Azmir Arifin, Navinash Nedumaran, Khairuddin Md Isa and Umi Fazara Md Ali

2516

Pertanika J. Sci. & Technol. 31 (5): 2505 - 2529 (2023)

effective, reducing the use of non-biodegradable mineral and vegetable oils (Japar et al., 2020). The selection of an effective thickener as a viscosity enhancer of base oil follows certain criteria, such as heat resistance quality, oxidation, and thermal stability. SBE has good potential as a grease thickener due to its high-temperature stability since SBE is made of mineral clay which does not melt easily (Abdulbari et al., 2011). Formulating grease with SBE results in grease with no dropping point exceeding 350°C can widely be used in high operating temperature applications (Abdulbari et al., 2017).

Apart from that, SBE is easily accessible as an industrial waste, well-known usage in palm oil refineries and food industries, which is costly to dispose of due to its high organic matter content. Disposal of SBE in landfills has the potential to cause pollution and fire hazards due to greenhouse gases emission and auto-ignition upon degradation of residual oil, respectively. The SBE waste generally contains residual, which quickly oxidises until spontaneous auto-ignition through autocatalysis reaction by the mineral clays. The SBE must be treated from its residual oil and undesired impurities prior to converting it into sustainable energy. An estimated 600,000 tonnes of SBE are discharged globally each year, with Malaysia, one of the largest palm oil manufacturers, disposing of approximately 195,000 tonnes in landfills (Cheong et al., 2013). Moreover, incineration and land disposal will likely be impossible soon due to stricter environmental regulatory restrictions and the lack of dumping sites. Recently, Malaysia has committed to becoming a carbon-neutral nation by 2050 which simply means balancing or reducing the emission of greenhouse gases such as  $CO_2$  (Susskind et al., 2020). Therefore, converting SBE waste as a thickener in WCO-based multi-purpose grease is an environmentally friendly, simple, and costeffective way to convert waste to wealth and carbon-neutral.

SBE, as a thickener in WCO grease formulation, holds a major problem in the mechanical stability of the grease. The SBE and WCO do not completely homogenise, causing oil separation and sedimentation during storage (Abdulbari et al., 2017). Thus, adding fumed silica as a modifier with SBE can improve the grease strength and stability during operation with a slight increase in the coefficient of friction at the accepted range (Abdulbari et al., 2017). Other advantages also include boosting the thermal stability of grease up to 150°C without degradation and having no effect on the corrosivity towards metal. A recent study also showed that the dropping point of grease comprised waste transformer oil (WTO) and sodium stearate thickener was enhanced from 150°C to 185–200°C, which is 3.6% using fumed silica (Rahman et al., 2020). The F.S. molecules bond in a strong interaction and form a new non-melting structure in the grease.

Furthermore, wear preventative additives such as the popular molybdenum disulfide and graphite are needed to boost the performance of the formulated grease. However, there is no evident research on the performance of these additives in WCO-based grease to compete with commercial grease in the market. Over the years, Molybdenum disulfide and graphite

have demonstrated satisfactory results as corrosion inhibitors and anti-wear additives in grease formulations. Early's earlier study (Sinitsyn & Viktorova, 1968) concluded that  $MoS_2$  has greater antifriction properties for Si and calcium-complex while denoting 30% more efficiency for Li grease than graphite.  $MoS_2$  has a layer lattice structure, and the atoms in each layer or "basal plane" are positioned at the corners of regular hexagons, leading to higher load-carrying capacity (Mohammed, 2013).

Meanwhile, combining MoS<sub>2</sub> with graphite at a 60:40 ratio has a synergetic effect in increasing the weld load by 120 kg while decreasing the wear scar diameter by 22% (Antony et al., 1994). Also, these combinations at any ratio increase the extreme pressure or wear characteristics of the organoclay grease type. Without a scientific study, this synergistic effect in MoS<sub>2</sub> and graphite will not work on WCO-SBE-based grease. MoS<sub>2</sub> is more efficient and economical than graphite in Li, Ca, Na and Li-Ca grease due to its fewer wear properties. In recent years, environmental concerns have necessitated focusing on renewable resource-based components for grease composition (Abdulbari et al., 2015). Thus, recycling waste cooking oil and SBE, which are disposed of freely after expensive treatment into the environment, will be a good option to address environmental issues such as greenhouse gas emissions, odour pollution and non-biodegradable. To enhance the performance of WCO/SBE grease, rheology modifiers such as fumed silica and additives such as molybdenum disulfide can be used widely with more research support in the future.

## The Treatments on Spent Bleaching Earth into Sustainable Energy

The treatment of SBE is crucial since it has sparked debate among researchers due to its hazardous properties. Many different methods are being applied to remove impurities in it. It is extremely costly within a large area requirement because SBE treatment does not only remove severe undesired impurities. Nonetheless, it can be converted into various valuable substances, including ash for the cement industry, industrial-grade oil, fatty acids, biodiesel, thickening agent, bio-fertiliser, and livestock feed.

A study by Chanrai and Burde (2004) revealed the general method of recovering oil from SBE. The recovery method was designed and invented by Narain Grirdhar Chanrai (Singapore) and Santosh Gajanan Burde (Johor). This method comprises three sub-process: reacting SBE with solvent between the temperature of 35°C and 50°C, separation of solid and liquid gained from slurry during previous steps, and extracting oil from the liquid obtained in the previous steps. The first treatment step begins with a reaction between SBE with solvent (n-hexane) from 35°C until 50°C in an agitated reactor for around 15 to 45 minutes. The main purpose of agitated reactor consumption is to dissolve oil from bleaching earth into the solvent. The standard acceptable range of slurry concentration inside the agitator's vessel is between 30% and 35%. Hence, the typical slurry produced will be further processed into a Porous Metal Filter (PMF) associated with plates to settle solids. This process is used to separate the solid from the miscella. Miscella is known as an oil solution in the solvent. PMF will process slurries under a range of 1 bar to 4 bars. This alternative method replaces gravity settlers' and the vacuum belt filter system. Usually, there are two stages of the filtration process for PMF to improve filtration efficiency.

As mentioned above, the first PMF has the output of miscella (being recycled to Miscella Distillation), and the separated cake comprises 70% to 75% dryness. The separated cake mixing of fresh or recycled hexane is carried out in the second agitated reactor. This process aims to recover the remaining or further adsorbed oil from the cake in the second agitated reactor vessel. The second PMF is further utilised as a separator between filtered cakes to produce Deoiled Bleaching Earth (DBE) with 3% to 5% of oil content through desolventisation. Meanwhile, severe miscella generated by the first PMF will undergo the distillation process to obtain extracted oil in the distillation plant. The distillation plant consists of a rising and falling film evaporator. The reaction may cause hexane to be produced, evaporated, condensed, and recycled back to the second agitated reactor. However, the extracted oil produced from the distillation unit is processed in deodorising units to remove free fatty acid (FFA). Other products, such as industrial-grade palm oil, will be yielded from this study.

The deoiled bleaching earth is from the desolventisation unit, resulting in less residue oil presence in processed SBE. So, the product of the resultant bleaching earth is considered safe to be disposed of in landfills due to minimum ignition possibility impact. Furthermore, it can be generated into anhydrous clay through the incinerator's burning process since it is a fuel substance (Afzan et al., 2020). Nonetheless, anhydrous clay is a good raw material that can be applied in cement manufacturing industries. Figure 4 shows the steps in treating SBE and turning it into useful products.

Smallwood (2020) discovered a method to reduce the fire ignition effect of SBE. It found that the fire explosion can be avoided by adding some Granular Salt or Brine at a specific rate, which allows the spontaneous combustion problem to be eliminated. To confirm this issue, Norman J. Smallwood also identified the hygroscopic effect properties of salt by exposing the solution (SBE + salt) under the sunlight during the summer of 2012 for one week. The solution did not ignite when the salt content was as low as 35% by weight. He proposed 45% as the best salt composition, defined as the minimum required content. The pattern of this salt formulation had been designed in the form of salt-lick products, either in block or granular form. It received extremely positive feedback from five cattle producers in the U.S. States of Colorado, Missouri, and Louisiana after testing. This treatment is most commonly used to convert SBE into animal and livestock feed supplements for further nutrition development.



Muhammad Auni Hairunnaja, Mohd Aizudin Abd Aziz, Nur Amira Fatihah Bashari, Mohd Azmir Arifin, Navinash Nedumaran, Khairuddin Md Isa and Umi Fazara Md Ali

Figure 4. Steps in treating SBE into sustainable products (Chanrai & Burde, 2004)

#### Selection of Fumed Silica (F.S.) as Thickener for Grease Formulation

Fumed Silica is also known as pyrogenic silica because it is produced by a flame reaction between silicon tetrachloride and oxygen (Ha et al., 2013). It is available in white powder form and has non-toxic properties. It is commonly used as a thickener and anticaking agent.

Moreover, it is made up of silicon dioxide with an amorphous shape (Rahman et al., 2018) and covered in silanol groups (Vansant et al., 1995). The silanol groups are extremely reactive and may be used to initiate chemical processes. The number of silanol groups present around the surfaces of F.S. powder determines its reactivity surface (Whitby, 2020). According to Barthel et al. (2005), the structure of F.S. is related to having space-filling particle properties against a large surface area. It is associated with a small number of micropores within a range surface area between 50 and 400 m<sup>2</sup>g<sup>-1</sup>. Therefore, it can be used as a free-flow additive in powder-like solids, forceful reinforcing filler in elastomers, and thickening in various liquids. In WCO, F.S. will clump together and create three-dimensional networks that can immobilise the oil by keeping its shape and constructing structural frameworks in solvents. It exists in nanoparticle form, with its surface chemistry and shape. The silanol groups on the particle surface work as molecular adsorption sites for species that establishes hydrogen bonds or interact with one another as donors or acceptors. According to Adhikari et al. (1994), the adsorption of triglyceride molecules in vegetable oils onto silica powder surfaces occurs as a result of hydrogen bonding formation between the silanol groups and the ester carbonyl groups. It will significantly reduce the silanol group that repeatedly forms hydrogen bonds. Israelachvili (2011) and Raghavan et al. (2000) stated that a 'Van der Waals' attraction is a key factor in the aggregation of F.S. in WCO. It may result in potential energy availability during mixing due to the electrostatic forces between the overlapping double layers of molecules (hydrogen bonding between silanol groups and adjacent hydrophilic

fumed silica particles). Then, it brings a semi-solid mixture forms without any leakages (Whitby, 2020).

The use of F.S. has numerous advantages. Its structure is in anisotropic network form, which resembles fats in form and mechanical properties. The network synthesis by F.S. in WCO increases the mixture viscosity and thickens structure stability (Whitby, 2020). Sugino and Kawaguchi (2017) mention that having an extra F.S. in oil can turn the liquid (oil) into gel form. Instead of thickening and anticaking agents, it can also be used as an anti-foaming and anti-blocking agent in catalysis, cosmetics, paper coating, pharmaceuticals, cable insulation, adhesive, construction, and automotive, and applied in cartridge toner for printing, stabilising agent in the food industry, bleaching cream and fire extinguisher. Therefore, selecting F.S. in grease formulation is a good idea due to its properties and physical structure, improving grease performance and meeting the industrial-grade criteria.

However, the issue regarding F.S. consumption is improper handling of this substance during operation, as Rav et al. (2020) mentioned. Several increment cases had been reported between 2015 and 2016 due to the respiratory problems caused by F.S. Commonly, Single-dose exposure to F.S. does not cause lung disease. Still, repetitive inhalation (multiple-dose) may cause a fibrogenic formation and bio-persistence process of F.S. in the lung (Sun et al., 2016). The foreign material will be present in the lung, and no treatments are currently available to remove F.S. from the lung. This problem causes patients to have difficulty breathing. The key challenges for the DOSH communities are raising awareness among stakeholders, workers, and even the public about the long-term effects of uncontrolled dust release and the magnitude of health problems caused by silicosis (Garcia et al., 2019). Therefore, proper precautions should be taken against F.S. during application. Table 4 summarises F.S. as the selected thickener for grease formulation.

Table 4

Summarisation on F.S. as a selected thickener for grease formulation

Full Name	Fumed Silica
Nomenclature	F.S.
Characteristic	<ul> <li>It is available in white powder form and has non-toxic properties.</li> <li>Made up of silicon dioxide with an amorphous shape covered in silanol groups.</li> <li>The silanol groups are extremely reactive and may be used to initiate chemical processes.</li> <li>The structure of F.S. is related to having space-filling particle properties with a small number of micropores within a range surface area between 50 and 400 m<sup>2</sup>g<sup>-1</sup></li> </ul>
Application	<ul> <li>against a large surface area.</li> <li>It is commonly used as a thickener and anticaking agent.</li> <li>It can also be used as an anti-foaming and anti-blocking agent in catalysis, cosmetics, paper coating, pharmaceuticals and cable insulation.</li> </ul>

Muhammad Auni Hairunnaja, Mohd Aizudin Abd Aziz, Nur Amira Fatihah Bashari, Mohd Azmir Arifin, Navinash Nedumaran, Khairuddin Md Isa and Umi Fazara Md Ali

Table 4 (continue)

Full Name	Fumed Silica
Advantages	<ul> <li>Its structure is in anisotropic network form.</li> <li>The network synthesis by F.S. in WCO increases the mixture viscosity and thickens structure stability.</li> </ul>
Hazardous	<ul> <li>Respiratory problems caused by F.S., according to several increment cases, had been reported between 2015 and 2016 (Rav et al., 2020).</li> <li>The repetitive inhalation (multiple-dose) may cause a fibrogenic formation and bio-persistence process of F.S. in the lung. No treatment is available to remove F.S. from the lung.</li> <li>It may accurate difficulties in breathing.</li> </ul>
Taking Action	<ul> <li>Raising awareness among stakeholders, workers, and even the general public about the long-term effects of uncontrolled dust release and the magnitude of health problems caused by silicosis is quite challenging.</li> </ul>

#### Molybdenum Disulphide as Selected Additives in Grease Formulation

 $MoS_2$  is a dark grey to black powder (Epshteyn & Risdon, 2010). It is widely used in a variety of applications outside the aerospace industry. Other reasons for its extensive applications are layer structure, large specific area, special electronic and electrochemical properties, and good surface modification properties (Cui et al., 2016). It has also been an excellent solid lubricant for years and was traditionally used as grease for bit lubrication (Fink, 2021). MoS<sub>2</sub> has a solid lamellar structure composed of atomically thin planes that easily slide between each other (Savan et al., 2006). The cross-sectional area for lamellar structure within the eutectic system comprises a free surface and solid-liquid interface, enabling the structure to move smoothly with less friction. The increased lamellar spacing may result in a slower solidification rate (Bei et al., 2003). It means that the grease with semi-solid form will provide larger lamellar spacing (larger solid-liquid interface), enabling it to provide less frictional force between grease and plane (surface) due to interface gaps availability that can fit with contact surface area. According to Savan et al. (2006), there is a significant difference between lubricants within and without MoS<sub>2</sub> addition, where it tends to improve the adhesion to the substrate, provide higher density and viscosity of grease, and extensively possess higher purity, which are the key factors of tribology improvement properties that include wear resistance enhancement and low coefficient of friction. A study by Srinivas et al. (2017) also revealed the effectiveness of MoS<sub>2</sub> nanoparticles as additives in lubricant technology to commercial oil (SAE 20W-40 grade), and the result showed that the nano lubricants reduced wear and friction when compared to commercial oil. Zebox et al. (2022) also found that adding MoS<sub>2</sub> in grease minimised the wear part of equipment by increasing the service life at the part of the frictional unit. Table 5 summarises MoS2 as the selected additive for grease formulation.

#### Grease Formulation from Palm Oil and Other Industrial Wastes

Full Name	Molybdenum Disulphide
Nomenclature	$MoS_2$
Characteristic	<ul> <li>It is a powder with a dark grey-to-black appearance.</li> <li>It has a good layer structure, a large specific area, special electronic and electrochemical properties, and good surface modification properties, which make it reasonable for extensive application.</li> <li>Comprises a solid lamellar structure.</li> </ul>
Application	• Widely used in a variety of applications outside the aerospace industry.
Advantages	<ul> <li>It also has been an excellent solid lubricant for many years and was traditionally used as grease for bit lubrication.</li> <li>Easily slide between each other due to the presence of solid lamellar structures with atomically thin planes.</li> <li>It makes the grease structure move smoothly with less frictional forces due to the free surface and solid-liquid interface by a eutectic system of lamellar structure.</li> <li>MoS<sub>2</sub> improves the adhesion to the substrate, provides higher density and viscosity of grease, and extensively possesses higher purity; all of them are the key factors of tribology improvement properties that include wear resistance enhancement and low coefficient of friction.</li> </ul>
Prove of Study for Lubricating within MoS <sub>2</sub> Application.	<ul> <li>Srinivas et al. (2017) also revealed the effectiveness of MoS<sub>2</sub> nanoparticles as additives in lubricant technology to commercial oil (SAE 20W-40 grade). They found that nano lubricants reduced wear and friction compared to commercial oil.</li> <li>Zebox et al. (2022) also found that adding MoS<sub>2</sub> in grease minimised the wear part of equipment by increasing the service life at the part of the frictional unit</li> </ul>

# Table 5Summarisation of MoS2 as a selected additive for grease formulation

#### **CONCLUSION**

SBE as a grease thickener is better due to its sticky properties, high viscosity and density. The viscosity of the mixture minimises penetration risk against WCO. Unfortunately, the study also found a tendency of SBE to cause spontaneous auto-ignition reactions due to the rapid oxidation of residue oil and autocatalysis action by clay minerals. This study shows that the SBE must first be treated to be free from residue oil and for safe use. According to Jong (2021), the minimum consideration for SBE as hazardous waste should be less than 3% of oil presence. The SBE also can be treated by adding (salt) to cut off the spontaneous auto-ignition reaction, as shown in Smallwood (2020). Using WCO as a base oil is a good idea because it is closely related to environmental protection quality and has a lower peroxide value. This waste source can be recycled to generate renewable energy. WCO should be filtered, heated, and stored in a tightly sealed container to reduce rancidity. Thus, the F.S. is recommended for use, but it must be properly managed, and continuous inhalation must be avoided during handling. MoS<sub>2</sub> is recommended to enhance grease performance as an antioxidant and heat-resistant agent. Wearing Personal Protective Equipment (PPE) is strongly advised to reduce the risk of exposure and silicosis disease.

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Muhammad Auni Hairunnaja, Mohd Aizudin Abd Aziz, Nur Amira Fatihah Bashari, Mohd Azmir Arifin, Navinash Nedumaran, Khairuddin Md Isa and Umi Fazara Md Ali

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Pertanika J. Sci. & Technol. 31 (5): 2505 - 2529 (2023)

2526

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Muhammad Auni Hairunnaja, Mohd Aizudin Abd Aziz, Nur Amira Fatihah Bashari, Mohd Azmir Arifin, Navinash Nedumaran, Khairuddin Md Isa and Umi Fazara Md Ali

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