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Synthesis of Grease from Waste Oils And Red Gypsum

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ABSTRACT

This paper presents a synthesis of grease from waste oils and red gypsum. The objectives for this study are to develop grease from different types of waste oils, red gypsum and to determine the best quality of grease based on different ratio of red gypsum and fumed silica. Different ratios of fumed silica and red gypsum were tested in this research to determine which ratio will formulate the best grade of grease based on National Lubricating Grease Institute (NLGI) number grade grease. The proportions of fumed silica and red gypsum involves are (80% red gypsum, 20% fumed silica), (60% red gypsum, 40% fumed silica) and (50% red gypsum and 50% fumed silica). Evaluation is carried out using American Society for Testing and Materials (ASTM) to characterize the new formulation of grease in term of penetration test, dropping point test, type of thickener and amount of oil separated. This test is important to find the best formulation of grease from waste oils and red gypsum, by comparing it with greases that available in markets. It was discovered that the best formulated grease has no dropping point even after 240°C of heating which proves that the grease has a stable structure at higher temperature. Then, the amount of fumed silica and red gypsum increases, oil separation decreases while the consistency of the grease increases. Grease properties are not only dependent on the composition of the base oil but also on the size and configuration of particles of the thickening agent. The more fumed silica contains in the grease, better the grease's quality. From the result of the experiment, grease was well made using base oil from silicone oil, following by waste emulsion. Smooth grease was made by using waste cooking oil, while grease cannot form when using used oil.

INTRODUCTION

Grease may be defined as a solid to semi-solid material consists of three major components that form lubricating grease. These components are base oil, thickener and additives. Most grease produced today use mineral oil as their fluid components. The thickener gives grease its characteristic as a sponge that holds the lubricant in place. Additives mostly are oxidation and rust inhibitors, extreme pressure, antiwear and friction-reducing agents (Jeremy, 2008). There a few of common greases which are calcium grease or lime grease, aluminium grease, lithium grease, sodium grease, silicone grease and others (Boehringer, 1992).

Grease is a viscous semi fluid, which adhere strongly to the packing material (Adhvaryu *et al*., 2005). The base oil and thickener package are the major components in grease formulations that exert considerable influence on the behaviour of grease. Many types and combination of thickener and base fluids, along with the supplemental structure modifiers and performance additives, give final grease formulation their specialty.

The use of liquid lubricant generally requires sealing of bearing against loss of lubricants. This sealing problem can often be simplified if lubricants are employed which resist the deforming effect of gravity. Grease functions as a sealant to minimize leakage, to keep out contaminants and acts to keep deteriorated seals

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effective. Other than that, grease is easier to contain than oil. In comparison, grease, is easily confined with simplified, less costly retention devices (Jeremy, 2008). Grease also can resist change in structure or consistency during service, maintain mobility under conditions of application and provide corrosion protection (Boucher and Jones). In daily applications, it has become increasingly important that a grease composition is able to provide adequate lubrication at high temperature and it retain its properties under shear. Besides that, grease can be used for machinery that runs intermittently or is in storage for an extended period of time and also can be used for machinery operating under extreme conditions such as high temperatures and pressures, shock loads or slow speed under heavy loads. Under these conditions, grease provides thicker film cushions that are required to protect and adequately lubricate (NLGI Lubricating Grease Guide, 1996).

There are three problems need to be considered in this project. The main problem is actually waste emulsion. In industrial situations, an emulsion is formed that can seriously endangers the environment. With varieties of oil pollution and miscellaneous characters of the pollution sources, largeness of discharge and high requirements for treatment, control of such wastewater is faced with huge difficulties and pressure (Xin and Wan, 2013). Another main problem is environmental pollution by red gypsum waste from industries. Disposal of red gypsum by land filling or storage of massive amounts of gypsum in large pieces of land due to the high cost of the waste's treatment, gives the negative effects to the environment. Not only that, cost also one of the main problems that need to consider. Even though, formulation of grease from red gypsum and waste oils are subjected because they are wastes and easily to get, but the formulation of grease from raw materials are very expensive.

The main purpose of this study is to develop grease from different types of waste oils as the base oil and red gypsum as one of the thickener that use to formulate the grease, which is something new to be discovered by using different types of waste materials as the raw material for this research. This new generation formulation of greases was tested by using an ASTM (American Society for Testing and Materials) testing to determine the grade classifications for the grease based on NLGI (National Lubricating Grease Institute) grade (Salvatore, 2003).

MATERIALS AND METHODS

Chemicals:

Four types of oils used as a base oil in this study which are recovered oil from waste emulsion, silicone oil, waste cooking oil and used oils were taken from cafeteria and car’s workshop. Red gypsum is an industrial waste from Huntsman Trioxide Sdn. Bhd and fumed silica was purchased from Sigma-Aldrich. Both were used as a thickener. Molybdenum disulphide as additive was purchased from Acros with a 98.5% purity. Three components that need to be mix to produce iron octoate paste that used as an anti-oxidant agent are octoic acid that purchased from Acros which has 99% purity, caustic soda with 96% purity was purchased from Merck and ferrous sulphate that also purchased from Merck, has 99.5% purity.

Sample preparation:

Formulation of Iron Octoate:

There are three steps involve in the process of iron octoate preparation. The first step is saponification process. Caustic soda is dissolve in 50ml deionized water. Then, octoic acid will be added with stirring and heating by hot-plate magnetic stirrer for two hours to produce soluble sodium soap. Temperature is maintained at 80-90oC. The second step is precipitation process. 200ml of 15% solution of ferrous sulphate is adding to precipitate iron octoate. The reaction time is 10 hours and the temperature is maintained. In the third step, drying process is involved. Slurry is filter and washed by vacuum filter, then dried in an oven at 65^{o}C for 90 minutes. The dried precipitations then crush into powders.

Formulation of Grease:

There are four steps involve in the formulation of grease. The first step is all the materials are prepared in the desired amount. The second step is preparation of smooth paste. The desire amount of fumed silica and red gypsum is slurred with ½ of the require amount of waste oils with a continuous stirring. Then, the third step is, continuous mixing and heating at a maintain temperature. Lastly, the forth step is addition and continuous mixing. The rest ½ of the waste oils, iron octoate and molybdenum disulphide add slowly into the smooth paste. The mixing and heating process continue for 6 hours. The grease is prepared for 1kg for each ratio.

Sample Analysis:

ASTM testing’s done to determine the stability, quality and the properties for general usages. The tests involve are penetration test (ASTM D-217), dropping point test (ASTM D-2265), oil separation test ASTM D-1742) and type of thickener used.

Penetration test (ASTM D-217):

Penetration test is to determine to which NLGI (National Lubricating Grease Institute) consistency grade of grease belongs. NLGI consistency grade number is in the range from 000 to 6. Greases with a higher number are firmer, tend to stay in place and are a good choice when leakage is a concern. Grease consistency is measured at 25°C after the sample has been subjected to 60 double strokes. After the sample has been prepared, a penetrometer cone is released and allowed to sink into the grease under its own weight for 5 seconds. The depth the cone has penetrated is then read, in tenths of a millimeter. The further the cone penetrates the grease, the higher the penetration result and the softer the grease.

Dropping Point Test (ASTM D-2265):

The dropping point of grease is the temperature at which the first drop of grease falls from the cup. It's happen when the thickener loses its ability to maintain the base oil within the thickener. This test is a qualitative indication of the heat resistance of grease on applications where a semi-solid lubricant is required. A small grease sample is placed in a cup and heated in a controlled manner in an oven. When the first drop of oil falls from the lower opening of the cup, the temperature is recorded to determine the dropping point. Dropping point is a function of the thickener type.

Oil Separation Test (ASTM D-1742):

Oil separation test determine the ability of grease to separate oil during storage. The sample of grease supported in a sieve 40µm 924 mesh), is subjected to 1.7kPa air pressure for 30 hours at 177°C. The quantity of oil separated through gauze cone is taken as a measure of the stability of the grease towards oil separation during storage.

Type of Thickener:

The grease formulated will be heated to relatively high temperature to determine whether the thickener within it is melting or maintain it structures. Basically, the grease that formulated using fumed silica as a thickener is categorized as a composed of non-melt thickener.

RESULTS AND DISCUSSION

Different ratios of fumed silica and red gypsum were tested with four types of base oils which were silicone oil, recovered oil from waste emulsion, waste cooking oil and used oil in this research to determine which ratio will formulate the best grade of grease. The proportions of fumed silica and red gypsum involves are (80% red gypsum, 20% fumed silica), (60% red gypsum, 40% fumed silica) and (50% red gypsum and 50% fumed silica). Evaluation is carried out using American Society for Testing and Materials (ASTM) to characterize the new formulation of grease in term of penetration test, dropping point test, type of thickener and amount of oil separated.

Silicone oils are primarily used as lubricants or hydraulic fluids. They are excellent electrical insulators and, unlike their carbon analogues, are non-flammable. Their temperature stability and good heat transfer characteristics make them widely used in laboratories for heating baths ("oil baths") placed on top of hotplate stirrers. Silicone oil also exhibits heat stability, oxidation resistance, very low vapour pressure, and high flash points. It is insoluble in organic liquid other than active solvents and is non-greasy, non-acidifying and virtually odourless (Martin, 1997). A silicone's molecular weight is a function of its degree of polymerization, the number of repeating Si-O-Si units in the polymer. When the degree of polymerization is high and the polymers are longer, then the viscosity will be higher. The higher the viscosity, the more slowly the polymer will flow. Viscosity has relatively little effect on the chemical properties of a silicone fluid, but it does affect flow behaviour and solubility.

Fumed silica serves as a universal thickening agent and an anticaking agent (free-flow agent) in powders. Fumed silica also known as pyrogenic silica because it is produced in a flame, consists of microscopic droplets of amorphous silica fused into branched, chainlike, three-dimensional secondary particles which then agglomerate into tertiary particles. The resulting powder has an extremely low bulk density, non-porous particles and high surface area. Its three-dimensional structure results in viscosity increasing, when used as a thickener. In this project, fumed silica used is aggregates type. Aggregates are collections of smaller particles that have been permanently joined together. In fumed silica, the primary particles have sintered together to form an aggregate, which is extremely strong. The aggregate is the smallest particle to which fumed silica can be dispersed (Garret, 1992).

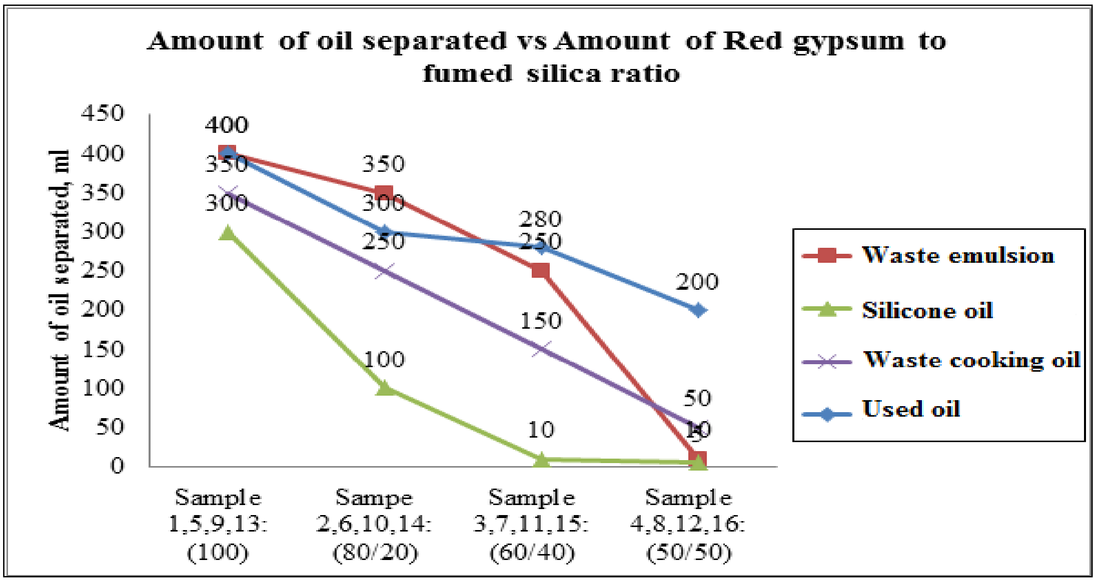

Fig. 1: Grease stability graph by four types of base oil.

The range temperature used during grease formulation is between 80-90°C of heating. Based on Figure 1, grease was well made using base oil from silicone oil, following by waste emulsion. Smooth grease was made by using waste cooking oil, while grease cannot form when using used oil. Grease properties are not only dependent on the composition of the base oil but also on the size and configuration of particles of the thickening agent. From experimental observations, it was clear that the amount of fumed silica affects the physical properties of grease. Not only that, less efficient of stirrer to break the particles into very fine forms during experiment also effect on the grease structure and appearance. The more fumed silica contains in the grease, the more its tendency to become solid form. The dispersive of fumed silica were not recognized in the grease because it was well mixed during the mixing process.

Grease Stability and Consistency Analysis:

After storage about two months, the grease samples are observed to test the stability. There were oil separation occurred in a few of the samples.

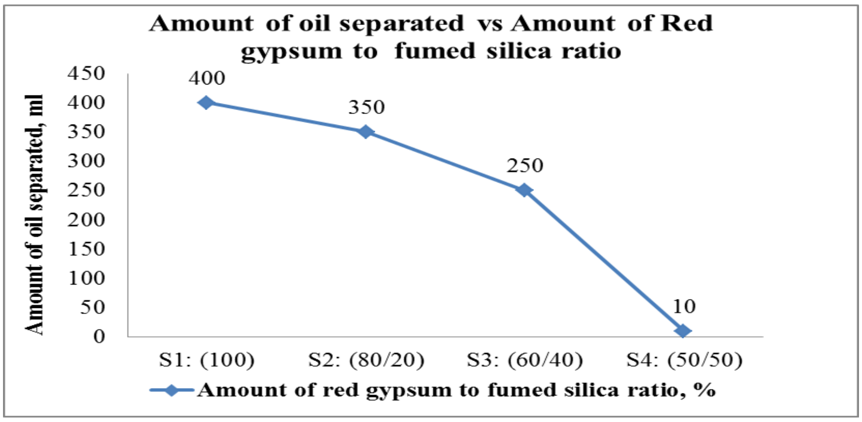

Fig. 2: Amount of oil separated for recovered oil from waste emulsions.

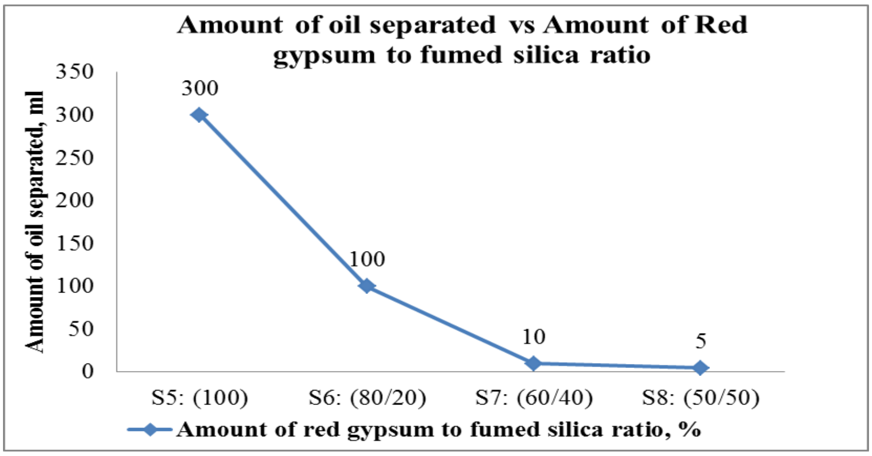

Fig. 3: Amount of oil separated for silicone oil.

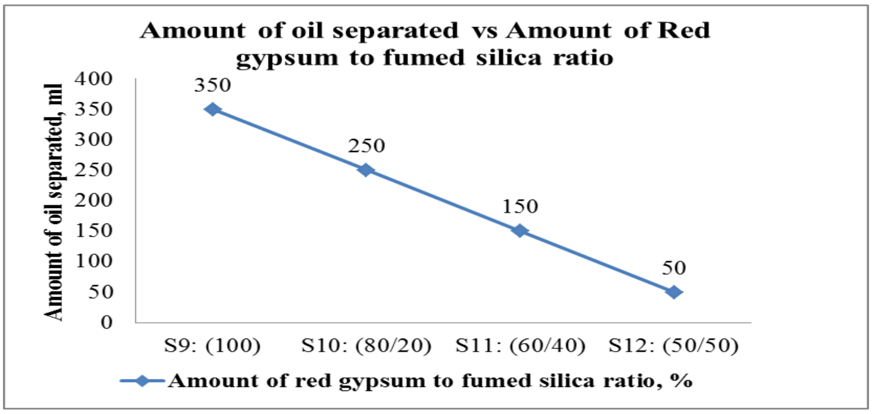

Fig. 4: Amount of oil separated for waste cooking oil.

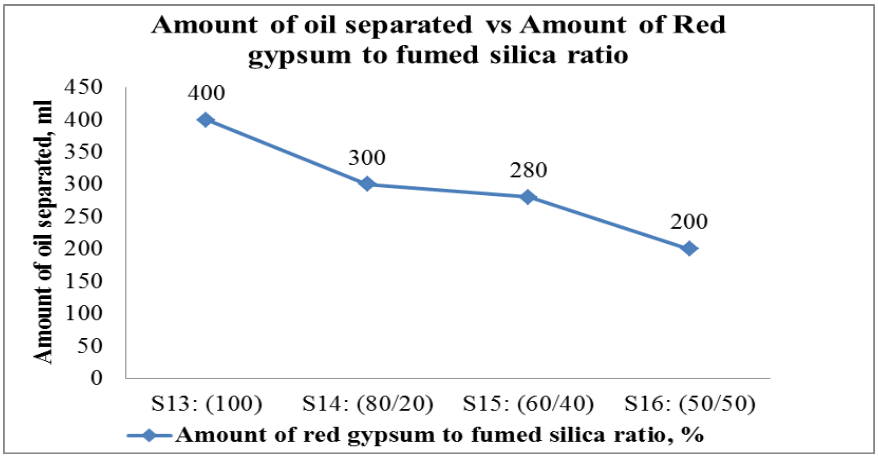

Fig. 5: Amount of oil separated for used oil.

From the figure 2, 3, 4 and 5 shown above, it's proved that by increasing amount of fumed silica percentage, the amount of oil separation will decreases rapidly. This situation occurred because due to the small percentage of fumed silica, results in deficient of strength in grease; resulting the grease loose the stability and the oil separation become higher. Fumed silica particles function as thickener is tends to hold and attract the oil together. The reduction amount of fumed silica results oil not stable in grease. After 50% wt. of fumed silica, oil separation becomes approximately invisible.

Physical Analysis on Best Formulation:

Table 1 shows the result after ASTM testing's that was conducted for the best formulated grease which is sample 4 (recovered oil from waste emulsion as base oil). This grease sample is semi-fluid with a dark-grey colour and has a rough appearance. There is also no melting even after 240°C of heating.

Table 1: Specifications of grease (Sample 4)

Test	Method	Result
Cone Penetration	ASTM D-217	340
Colour	ASTM D-1500	Dark grey
Appearance	ASTM D-4176	Rough
Dropping Point	ASTM D-2265	No Melting after 240°C
Worked 60x Stroke	ASTM D-217	295
NLGI Grade		1

The dropping point of this sample is above 240°C; shows that the formulated grease are stable and can retain heat resistance at high temperature. Dropping point also indicates the upper limit which the grease can retain their structure. Addition of 50% fumed silica and 50% red gypsum does not influence the stability of grease, then it's applicable to use at higher temperature. As the amount of fumed silica increases, the consistency of grease transforms from semi fluid to very soft. Grease consistency depends on the type and amount of thickener used and the viscosity of base oil. Grease's consistency is the resistance to deformation by an applied force. The consistency stability for formulated grease is satisfactory at 50% fumed silica amount.

Conclusions:

This research proved that grease can be formulated by using waste oil as the base material By screening through all the greases formulated, the waste emulsion based grease (sample 4) was recorded to be the most stable grease. The grease formulated had shown overall good performances from the ASTM standard method of testing. The grease formulated was able to work at high temperature condition which was proven by the dropping point of 240°C. Besides, the grease formulated had low penetration number, indicates that the consistency of the grease can be described as very firm appearance. Whereas for the oil separation test, there was only little amount of oil separated from the grease itself. Thus, it can be concluded as the amount of thickener affected the consistency as well as the stability of the grease. Fumed silica was able to work as the thickening agent in the formulation of grease from waste oil) and it showed overall good performances.

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