FORMULATION OF MULTI PURPOSE GREASE FROM TITANIUM DIOXIDE MANUFACTURING WASTES (RED GYPSUM)

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I declare that this thesis entitled "Formulation of Multi Purpose Grease From Titanium Dioxide Manufacturing Wastes (Red Gypsum)" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ……………………………………..

Name : ……………………………………..

Date : ……………………………………..

Special Dedication of This Grateful Feeling to My Beloved father and mother;

Mr. ARUMUGAM S/O THAMBUSAMY and Mrs. SARASWATHY D/O RATNAM

Loving brothers;

A.KALIDAS, A.JAGATHISAN AND A. SARAVANAN

Supportive families;

Uncles and Aunties

For Their Love, Support and Best Wishes.

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ABSTRACT

This study describes the formulation of multipurpose grease using red gypsum from titanium dioxide manufacturing wastes. Greases produced with different amount of fume silica (FS) and red gypsum in its compositions. The effect of red gypsum's and fumed silica's concentrations on the formulated greases has been studied. The grease mixture is formulated using silicone oil as base oil, fumed silica and red gypsum as thickener, molybdenum disulphide as additive and iron octoate as anti-oxidant agent. Amount of fumed silica involves 2-20% wt (2% wt, 7% wt, 16% wt and 20% wt). All the fume silica proportions covered (70% gypsum, 30% FS), (60% gypsum, 40% FS) and (50% gypsum, 50% FS). The testing (penetration test, type of thickener, dropping point test, and amount of oil separated) have been done on the formulated greases to determine the properties. It was discovered that formulated grease has no dropping point until 260 ^{o} C which proves that the greases has stable structure at higher temperature. All the formulated greases proved to be composed of non-melt thickener type. As the amount of fumed silica and red gypsum increase oil separation decreases and the consistency of the greases increases. The penetration also significantly decreases as fumed silica addition increases. This is due to the larger strength and small size of fumed silica. The larger strength of fumed silica was thought to be due to the structure being knit together by means of primary valence bonds. It can be concluded that the formulated grease is multi purpose grease. Higher the amount of fumed silica and gypsum (thickener), better the grease's quality. Therefore, the formulated greases has high potential to be used in wide range of industries such as the cars, ships, aircrafts and most of the industries that deals with heavy duty equipments.

ABSTRAK

Kajian ini menggambarkan penghasilan gris serbaguna menggunakan gypsum merah daripada bahan-bahan buangan dalam menghasilkan titanium dioksida. Gris dihasilkan daripada kuantiti serbuk silika halus ('fumed silica') dan gipsum merah yang berlainan. Kesan perubahan kuantiti gipsum merah dan serbuk silika halus pada gris yang dihasilkan telah dikaji. Gris dihasilkan menggunakan minyak silikon sebagai minyak utama (primer), serbuk silika halus dan gipsum merah sebagai agen pemekatan, molibdenum disulfida sebagai bahan penambah dan ferum oktat ('iron octoate') sebagai agen antioksida. Serbuk silika halus digunakan dalam kadar 2-20% berat (2% berat, 7% berat, 16% berat dan 20% berat). Kesemua berat itu meliputi (70% gipsum, 30% serbuk silika halus), (60% gipsum, 40% serbuk silika halus) dan (50% gipsum, 50% serbuk silika halus). Beberapa eksperimen (ujian penembusan, jenis agen pemekatan, ujian menentukan suhu dimana gris berubah menjadi cecair dan kuantiti minyak yang berpisah) telah dilakukan pada gris yang dihasilkan untuk menentukan ciri-cirinya. Ia telah dibuktikan bahawa gris yang dihasilkan tidak berubah menjadi cecair sehingga suhu 260 ^{o}C yang membuktikan gris ini mempunyai struktur stabil pada suhu yang tinggi. Kesemua gris yang dihasilkan dibuktikan mengandungi jenis agen pemekatan yang tidak bercair. Sekiranya kuantiti serbuk silika halus dan gipsum merah bertambah, kuantiti minyak yang terpisah dari gris berkurang dan konsistensi gris pula meningkat. Nilai penembusan juga menurun apabila penambahan serbuk silika halus semakin bertambah. Ini disebabkan oleh saiz serbuk silika halus yang kecil menyebabkan kekuatan yang tinggi. Kekuatan tinggi serbuk silika halus disebabkan oleh struktur yang dijalin bersama dari ikatan valens primer. Secara kesimpulannya, gris yang dihasilkan adalah gris serbaguna. Semakin tinggi kuantiti serbuk silika halus dan gypsum (agen pemekatan), semakin baik kualitinya. Oleh itu, gris yang dihasilkan mempunyai potensi yang tinggi untuk digunakan dalam pelbagai sektor seperti kereta, kapal layar, kapal terbang dan juga industri yang menggunakan alat-alat yang bertenaga tinggi.

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LIST OF ABBREVIATIONS

FS	-	Fumed Silica
wt	-	weight
DI	-	deionized water
m	-	mass
sg	-	specific gravity
g	-	gram
%	-	percent
ml	-	milliliter
w	-	weight
V	-	volume

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CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Grease is a semi fluid to solid mixture of a fluid lubricant which would normally have been employed, together with a thickener, additives and anti-oxidant agent. The fluid lubricant that performs the actual lubrication can be petroleum (mineral oil), synthetic oil, or vegetable oil. The thickener gives grease its characteristic consistency and is sometimes thought of as a "sponge" that's holds the oil in place. Additives enhance performance and protect the grease and lubricate surfaces. The most common grease are calcium grease or lime grease, sodium grease, aluminum grease, lithium grease, silicone grease, polyurea grease and others (Boehringer,1992).

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. Such "solidified" lubricants are usually called "greases". Grease is a viscous semi fluid, which adhere strongly to the packing material. The two basic structural components of grease are: a thickening agent and liquid or base fluid, in which that thickening agent is dispersed. Many types and combination of thickener and base fluids, along with supplemental structure modifiers and performance additives, give final grease formulation their specialty. Many types of thickening agents are being added to both petroleum oils and synthetic oils to formulate grease, which are usually soap-thickened mineral oils to which the different soaps employed as thickeners give different texture. Some of more common thickening agents are non-soap-thickening agent as bentonite (Adhvaryu *et al*., 2005 and Boner, 1954).

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 that it is subjected to during use. Besides that, grease functions as a sealant to minimize leakage and to keep out contaminants. Grease can be used for machinery that runs intermittently or is in storage for an extended period of time and also for machinery operating under extreme conditions such as high temperatures and pressures. Under these conditions, grease provides thicker film cushions that are required to protect and adequately lubricate (NLGI Lubricating Grease Guide, 1996). Perhaps one of the most contributions of grease industry has been to the aircraft industry, particularly the military aircraft equipment. There is an increasing demand for lubricants in many fields, such as in military applications, with high load carrying capacity and very high operating temperature, as well as very low temperature specified. These are requirements that give new significance to certain properties such as the thermal stability and relative indifference to temperature extremes.

The present research deals with formulation of multipurpose grease from titanium dioxide manufacturing wastes to use wherever severe operating conditions are encountered. This research includes the study of the effects of thickening agents (gypsum wastes and fumed silica) on physical properties besides addition of additive (molybdenum disulfide, MoS_2) and anti-oxidant agent (iron octoate) in which iron octoate was prepared from octoic acid, ferrous sulfide and caustic soda using precipitation process.

1.2 RESEARCH BACKGROUND/PROBLEM STATEMENT

1.2.1 Objective

In the course of completing this project, there are few objectives. The objectives of the study are as follows:

1. To formulate multi purpose grease from titanium dioxide manufacturing waste (gypsum).
2. To determine the characteristics of formulated grease based on effects of fumed silica and gypsum's amount.
3. To monitor the amount of oil separated during storage.

1.2.2 Scope of Study

The scope of study will involve utilization of titanium dioxide manufacturing wastes (gypsum) to formulate multi purpose grease using batch method. Characteristics of formulated grease were analyzed based on the effect of fumed silica and gypsum's amount by performing conventional ASTM (American Society of Testing and Materials) methods. The performed tests were penetration test, dropping point test, type of thickener and amount of oil separated.

1.2.3 Problem Statement

The main purpose of this study is to formulate multi purpose grease from titanium dioxide manufacturing wastes (gypsum) which is something new to be discovered. There are many common types of grease are available in the market at this moment. But this is the first time where greases formulated from titanium dioxide manufacturing wastes (gypsum).

There are two problems in the considerations of solving in the course of this project. The main consideration is actually cost. Formulations of grease from traditional raw materials are expensive. Therefore, formulation of multipurpose grease from gypsum is subjected. In that case, gypsum from industry waste will play an important role as a source for this process.

Secondly environmental pollution will be the other problem to be solved. Disposal of gypsum by land filling or storage of massive amounts of gypsum in large pieces of land due to high cost of the wastes' treatment will give negative effects to the environment. Therefore, the present study converts the negative cost of treating the wastes to positive income to the industry by converting the wastes to valuable and usable product like grease.

CHAPTER 2

LITERATURE REVIEW

2.1 GREASE

Lubricating greases are usually made from oils thickened with metal soaps. In addition, lubricating greases usually contain some performance additives (Gow *et al.*, 1997). Therefore, the usage of polymers is a common practice to modify the rheological properties of greases by reinforcing the role of the thickening agent (Al-Sammerral, 1986).

The basic definition of Lubricating grease as mentioned by Jolicoeur (1992) is a semisolid product resulting from the fine dispersion of a thickening agent in lubricating oil. Grease consists of two basic structural components: A thickening agent and liquid or base fluid, in which that thickening agent is dispersed. Many types and combination of thickener and base fluids, along with supplemental structure modifiers and performance additives, give final grease formulation their specialty (Lubrizol Corporation, 2000).

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. Such solidified lubricants are usually called grease and are mostly composed of lubricating oil, which would normally have been employed, together with a gelling agent, which lends stiffness to the mixture. The essential property which grease must possess is the ability to form a film between surfaces so that surface- to-surface contact is prevented. This film may be quite thick, where grease is being used as an anti-friction bearing (Grandou and Masson, 1996). During the past few years, several investigators did their work on improving and studying the properties of the greases used in lubrication in the industry.

Generally, fume silica is a simple mixture of approximately 8% of fume silica gel with non-polar oil will yield grease of good consistency but with a matte surface. Greases based on organosiloxane fluids (such as poly methylsiloxane) thickened with fume silica are stabilized on storage and against the effects of heat. Fume silica particles (thickening agent) hold the fluid by adsorption (Avci, 2003 and Murray, 2000).

Electron micrographs of silica bodied lubricating fluids show aggregates of more or less rounded particles of the thickening agent. The adsorption of fluid by myriad of such particles may be responsible for holding the fluid when fine silica is used for thickening lubricating oil. Fume silica greases are useful in special high temperature application (Billelt, 1979). The upper limits are for the thickeners are determined by phase transformations of thickener, which cause modification in the grease structure. However, the presence of mineral oil limits their upper temperature use to approximately 200°C.

New generation of grease consisting more than one additive in order to increasing the performance of grease was developed. Some of the additive may contain grafted anhydride functional groups being progressively introduced in grease formulations (George and Scharf, 1996). In this present work, load-carrying and anti-oxidant chemicals were formulated and added as additives to achieve that purpose. Mixing efficiency is important during the whole manufacturing process (Scharf *et al.*, 2001). The high temperatures achieved during processing may significantly increase the reactivity of repeating unit of polymethylhydrogensiloxane oil (Verstrate and Struglinski, 1991).

It was proven that the addition of graphite or MoS_2 (8 in each case) improves the anti-seize and antifriction properties of greases in sliding friction. The effectiveness of such additives depends to a great extent on the chemical composition of the grease (Boner, 1954). Results have been presented previously from determinations of anti-wear and extreme pressure properties of polytetrafluoroethylene lubricating greases (PTFE greases) and dispersion media of various chemical types; in this work, the PTFE thickener was shown to be highly effective in friction.

Recently, many researchers tried to produce optimum performance for the grease. Yeong *et al.,* (2004), studied the steady flow and visco-elastic properties of lubricating greases containing various thickener concentrations. The flow properties show that grease is a shear-thinning fluid with a yield stress that increases with thickener concentration. Finally, the experimental results showed that, zero shear viscosity was also calculated from the creep experiment and as a result viscosities over nine orders magnitude were obtained. The power law index of the scaling law of the elastic modulus and yield stress with increasing volume fraction was found to be around 4.7 suggesting that the flocculation of the particles that compose the grease is likely to be of the chemically limited aggregation variety.

There also have some research to produce grease from certain byproduct like recycled LDPE, that Alfonso *et al*., (2007), studied the feasibility of recycling poly-olefins as additives to improve the rheological properties of lithium 12-hydroxystearate lubricating greases. The effects that both soap and recycled low-density polyethylene (LDPE) concentration exert on the rheology of lithium lubricating greases and its relationship with grease microstructure are discussed in this work. In that way, different lubricating grease formulations were manufactured by modifying the concentration of lithium 12-hydroxystearate and content of recycled LDPE, according to a RSM statistical design.

According to E. Armstrong (1955), grease can be formulated in either a batch or continuous process. Batch production is the most common manufacturing method. Bulk ingredients are metered or weighed into the processing reactor. For soap-based greases made by saponification, the fatty ingredient, alkali and a portion of the oil are added to the reactor. Heating and mixing convert the fat to soap, and the soap is dispersed throughout the mixture. Remaining oil is added to the batch after completion of saponification and dehydration to lower the temperature. Next the grease is milled and homogenized which will produce a uniform crystal and gel structure that will not change when the grease is used. The grease is further cooled, deaerated and packaged.

Adhvaryu *et al*., (2005) discussed the preparation of lithium soap-based soy greases using different fatty acids and the determination of crystallite structure of soap using Transmission Electron Microscopy (TEM). Lithium soaps with palmitic, stearic, oleic and linoleic acids were synthesized and mixed with Soybean Oil (SBO) and additives to obtain different grease compositions. His TEM measurements have revealed that the soap crystallite structure impact grease consistency. Soap fiber length and their cross-linking mechanism in the matrix control grease consistency (National Lubricating Grease Institute (NLGI) hardness, ASTM D-217 method). They showed that Lithium stearate-based soy grease has a relatively more compact fiber structure than Lithium palmitate. Linoleic acid with two sites of C-C in saturation in the chain has a much thinner and more compact fiber network than oleic acid (CI 8). The presence of additive in grease produces soap with looser network and larger fiber structure than similar grease containing no additive.

Franco *et al.,* (2005) evaluated the manufacture of lubricating greases through the mixing rheometry technique, by studying the effect of some processing variables such as rotation speed, intensity and duration of the homogenization treatment and thermal profile applied, in terms of the power-draw characterization and rheological behavior of the final product. With this aim, their lithium lubricating greases were prepared by inducing the saponification reaction between 12-hydroxystearic acid and hydrated lithium hydroxide within a naphtenic lubricating oil medium in an open vessel. The saponification reaction occurred until neutralization by stirring with a controlled rotational speed mixing rheometer using an anchor impeller. Different rotational speeds were selected. Finally, a highly intensive homogenization treatment was applied using a rotor-stator turbine in order to reduce crystal sizes.

Different homogenization treatments and cooling profiles were applied on the incipient greases. The American air force desired a lubricating grease to operate for several hundred hours at 204-232°C and yet be suitable for use at temperatures as close as possible to -53°C. Lubricating greases were therefore made, using silicon oil with a flashpoint of 316°C and pour point of -46°C. When operated in bearing at 232°C, a product containing 10% of silica as a thickener failed in 13 h and this is the applications of silica thickened greases have been used in aircraft. Number of investigators has made claims for the use of silica and other materials for thickening lubricating fluids. Fume silica act as a thickening agent in a liquid lubricant and form grease depends on its ability to remain suspended and to exert inter-particle forces, which will keep the system in relative equilibrium. The nature of these forces depends in turn on the type of thickening agent-employed.

2.2 RHEOLOGICAL CHARACTERISTICS OF GREASE

Greases are plastic non-Newtonian lubricants, i.e. there is a minimum value of applied stress called the yield stress or yield value, which is required to cause flow. While at higher stress they behave as viscous fluids, the viscosity falling rapidly with increasing stress and tending finally to the viscosity of the fluid component (Bogie and Harris, 1966).

A factor which greatly influences grease structure, is amounting of shear it under goes in a bearing. Effect of shear, or working of the grease, is to cause break down of the structure with the result that stiff grease may assume the properties of lower consistency grease (Krista Siik and Jyrki Vuorinen, 2005). Greases should therefore be of good mechanical stability. As the rate of shears increases, the apparent viscosity falls progressively as a result of break up of the aggregates. Under very severe conditions of shear and turbulence, as in the track of ball- or roller-bearing, the individual crystalline fibers may themselves break into smaller one.

The temperature of use the grease also grossly affects the structure. The effect of temperature upon viscosity is notably greater than it is upon other common physical property. If the temperature is high enough, the grease will under go a transition form the semi-solid to the liquid state, with complete structure breakdown. It is therefore essential that soap thickened greases are used at temperature well below their drop points. The change in the soap-crystallite-oil relationship with rise in temperatures, which differ for different soaps and oils, produce rheological effects, which may determine the quality of their performance in bearings (Marjorie, 1956).

If soap-based grease is heated, its penetration increases only very slowly until a certain critical temperature is reached. At this point the gel structure breaks down, and the whole grease becomes liquid. This critical temperature is called as dropping point of grease. Grease heated above its dropping point and then allowed to cool it usually does not fully regain its grease-like consistency, and its performance subsequently will be unsatisfactory (Horace, 1909). Therefore, it follows that at not time should the dropping point be exceeded.

The ideal grease would be one, which did not exhibit a change of structure or consistency as the temperature changed. When the possible changes due to temperature are considered it can be seen that often such change limit the temperature range over which a particular product can be used. However, when lubricating fluids are thickened with silica gel, carbon black or clay base products, phase change due to temperature will not take place.

2.3 COMPOSITION OF GREASES

Basically, red gypsum is extracted from ilmenite (a type of sand) ores before being refined into titanium dioxide (TiO_2). Red gypsum contains mainly hydrated $CaSO_4$ (70% wt), Fe_2O_3 (30%wt) and very small amount of Al_2O_3 (Rose Aini Kamarudin, 2007). The two main processes involved are chloride process (uses HCl) and sulphate process (uses H_2SO_4). There are two types of gypsum exist are natural gypsum and synthetic gypsum. Generally these gypsums are used in many applications in the industry such as plasterboard manufacture, cement manufacture, growing medium for mushroom, and soil conditioner. Mixing process throughout the whole process of multipurpose grease formulation is important (Franco *et al.*, 2005 and Scharf *et al.*, 2001). The reactivity of components towards high temperature will significantly increase to produce good product.

The most common oils used in grease formulations are petroleum oil, synthetic oil, vegetable oil, lubricant oil and etc (NLGI, 1999). In my research, silicone oil choose to used as base oil for grease due to its lubricant properties and also it tends to mix better with soaps and additives and form stronger structure.

Recycled byproduct like Low Density PolyEthylene (LDPE) also used to produce greases (Alfonso, *et al.,* 2007). This study shows that, feasibility of recycling poly-olefins as additives to improve the rheological properties of lithium 12-hydroxystearate lubricating greases. The results showed that both soap and recycled LDPE amounts exert in the rheology of lithium lubricating greases and its relationship with grease microstructure.

Basically, fumed silica, also known as pyrogenic silica, is a non-crystalline, fine grain, low density and high surface area silica. Fumed silica has a very strong thickening effect. It serves as a universal thickening agent. Besides fumed silica, other thickener that has been used widely in grease formulating industry is soap thickener which produced from fatty acids and alkali (NLGI, 1999).

Molybdenum disulfide is the inorganic compound which is non reactive, being unaffected by dilute acids (Fischer *et al.*, 1982). In terms of its appearance and feel, molybdenum disulfide is similar to graphite and indeed it is widely used as a solid lubricant due to its low friction properties, sometimes to relatively high temperatures. Usage of additives for this multipurpose grease formulation will increase the performance of grease (R Holinski, 2000). Other example of additives that can be used is graphite in certain applications at high temperatures.