## **FINAL REPORT OF GRANT UIC 161004**

# HIGH PERFORMANCE GREASE FROM INDUSTRIAL WASTE

NAMA FAKULTI : MOHD NAJIB BIN RAZALI : FAKULTI KEJURUTERAAN KIMIA & SUMBER ASLI

# **LIST OF CONTENTS**

- 1. Grant UIC 161004 Final Report
- 2. ORCA GreenTech Market Validation Agreement
- 3. MTDC Offer Letter ORCA 1
- 4. MTDC Offer Letter ORCA 2
- 5. Patent Base Oil 2016400017
- 6. Patent MNRg-Grease 2017400003
- 7. SDS MNRg-Grease
- 8. MNRg-Grease Quotation (130kg)
- 9. MNRg-Grease Invoice (130kg)
- 10. MNRg-Grease Delivery Order (130kg)
- 11. Front Page TNBr Grease Consultation Project
- 12. MNRg-Grease Consultant Quotation
- 13. MNRg-Grease Consultant Invoice
- 14. Consultancy Claim Report
- 15. Journal 1 AJBAS
- 16. Journal 2 IOP
- 17. Journal 3 IJET
- 18. Journal 4 Egypt Petroleum
- 19. MNRg-Grease Brochure

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# PROJECTS INFORMATION

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#### **PROJECTS SUMMARY**

Title:

## MNRg-Grease: High Performance Grease for Industrial Applications



G-Grease: High Performance Grease

MNRg-Grease is a high-performance grease was developed since 2012 by Mr Mohd Najib Razali and his team. It is formulated at low cost, environmental friendly and safe. This project was funded by CRDF1 under Malaysian Technology Development Corporation (MTDC) with amount of RM 271,000.00. This fund was acquired under Symbiosis 3.0 Programme with Orca Greentech Plt. CRDF is for funding of commercialization activities of locally developed technologies undertaken by Malaysian – owned companies. MNRg-Grease is readily available contact grease for the maintenance teams and also has been proven to be suitable for mechanical equipment. Stability, quality and properties of the product have been tested using ASTM (American Society of Testing and Materials) test. MNRg-Grease had been purchased with amount of **RM 34,400.00** for 160kg. For the consultation work, the amount is **RM 31,800.00**  1- State the name of the project involved and the duration of the project.

MNRg-Grease: High Performance Grease for Industrial Applications (Duration: 27<sup>th</sup> September 2018 – 26<sup>th</sup> September 2019)

2- State the objectives and background for each project.

Lubricant grease is used in any type of machinery with rolling and sliding contacts. These can include bearing, gears, couplings, slides, chains, pin and bushing points, hinges, constant velocity joints and etc. In manufacturing industry, there are a lot of imported and expensive grease used for machineries for it to function accordingly. It is common in industry that these machines are damaged due to unsuitable grease used. Even worse, some grease in the market right now might cause dermatitis (skin disease) due to inappropriate handling.

Grease is made up of three main elements which are base oils, thickener and additives. In MNRg-Grease, the thickener is derived from industrial waste.

Patent has been filed for area of usage of the waste material and its formulation in the product (Patent filing number: PI2017400003). Novelty claimed in the patent lies on the usage of combination of thickening agent for grease formulation. The grease to be manufactured varies in specification as per market needs. However, the key-component i.e. thickening agent is as per the patent filed.

MNRg-Grease is invented to be high performance grease that suits industrial use which includes electrical and mechanical appliances. It is also specially formulated with no carcinogenic elements. Currently, MNRg-Grease is manufactured in a laboratory scale at UMP with capability of 15 kg per day.

#### 1.High performance

Stability, quality and properties of the product have been tested with regulatory standards in market; ASTM (American Society for Testing and Materials) and NLGI (National Lubricating Grease Institute). Furthermore, qualitative analysis had been made with nine (9) greases at market and the result shows that MNRg-Grease has a performance that can compete with others.

#### 2. Safe.

MNRg-Grease will not cause skin rashes and other skin diseases when in contact with human. As reported by Yen Loo Lim and Anthony Goon, 2007 grease are one of the main factors which causes dermatitis among the workers (technician).

#### 3. Good Conductivity

MNRg-Grease is designed with good conductivity criteria. The product allows electricity to pass through with minimum number of resistance. Thus, MNRg-Grease helps in improving efficiency of the electrical appliance especially with switch gear.

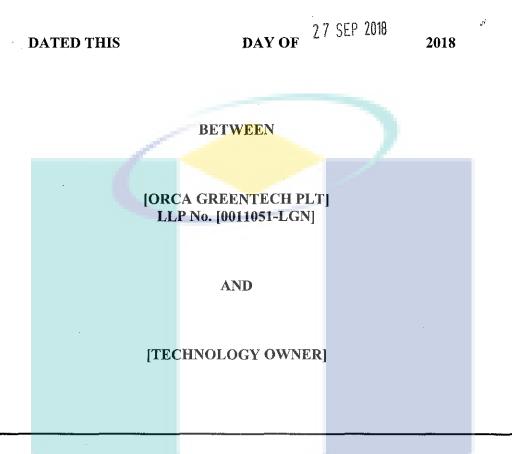
4. Waste to wealth.

The product is manufactured by utilizing used engine oil or industrial waste. The waste is used for the main ingredient to make the MNRg-Grease and it give the lubricating properties to the grease. Thus, productions of MNRg-Grease use less usage natural resources.

- 3- State the achievement of high impact projects. State also how industry / community partners have contributed towards achieving the objectives, impact and success of the projects.
  - 1. MTDC-CRDF 1 2017 (Commercialization of Research & Development Fund RM271,100.00) with Orca Green Tech Pvt. Ltd.
  - 2. Gold Medal, Creation, Innovation, Technology & Research Exposition (CITREX 2019) UMP, Malaysia.
  - 3. Silver Medal, International Exhibition Ideas-Invention-New Products (IENA 2015), Nuremberg, Germany.
  - 4. Gold Medal, 25th International Invention, Innovation & Technology Exhibition (ITEX 2014).

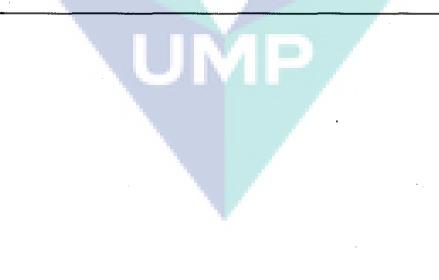
List of Clients

- Orca GreenTech Pvt.Ltd
- TNB Research Sdn Bhd.
- 4- State the form of income generated in the form of cash or in-kinds
   Explain:
   Manpower contribution or financial contribution / significant in kind
  - 1. MTDC-CRDF 1 2017 (Commercialization of Research & Development Fund RM271,100.00) with Orca Green Tech Pvt. Ltd.
  - 2. MNRg-Great Sales (160 kg) RM 34,4000.00
  - 3. MNRg-Grease Consultation Work -RM 31,800.00
  - 4. Consultation Work for TNB Research Sdn. Bhd 2016 (Chemical Testing and Analysis on Electric Contact Lubrication for Middle Voltage Switchgears) RM RM119,780.00
- 5- History of industry / community network prior to high-impact projects along with industry / community partners nominated.
   Explain: sharing of expertise / knowledge that enhances project impact to the community.
  - 1. Malaysia Technology Development Corporation (MTDC)
  - 2. Orca GreenTech Pvt LTD
  - 3. TNB Research Sdn Bhd.



#### **TECHNOLOGY AND MARKET VALIDATION AGREEMENT**

RELATING TO THE TECHNOLOGY AND MARKET VALIDATION OF [HIGH PERFORMANCE GREASE USING RECYCLED WASTE OIL



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## TABLE OF CONTENTS

	RECITALS	[]
1.	DEFINITION & INTERPRETATION	[]
2.	CONDITION PRECEDENT	[]
3.	GRANT OF RIGHTS	[]
4.	RESPONSIBILITIES OF THE TECHNOLOGY OWNER AND THE PARTNERSHIP	[]
5.	TERM AND EXTENSION OF TERM	[]
6.	RIGHT TO OBTAIN LICENCE	[•]
7.	IMPROVEMENTS	[]
8.	FINANCIAL PROVISIONS	[]
9.	CONFIDENTIALITY	[]
10.	NON-COMPETITION	[]
11.	TERMINATION	[]
12.	REPRESENTATION AND WARRANTIES	[]
13.	INTELLECTUAL PROPERTIES PROTECTION AND	[]
	PROCEDURES	
14.	COSTS	[]
15.	COMMUNICATIONS	[]
16.	DISPUTE RESOLUTION COMMITTEE	[]
17.	NATURE OF AGREEMENT	[]

e.

2

SCHEDULE I	[]
SCHEDULE II	[]
SCHEDULE III	[]
SCHEDULE IV	[]

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¢,

THIS TECHNOLOGY AND MARKET VALIDATION AGREEMENT (the "Agreement")is made thisday of201[].

#### BETWEEN

The Partnership, particulars of which are set out in Schedule I of one part

AND

The Technology Owner, particulars of which are set out in Schedule I of the other part.

(The Partnership and the Technology Owner where the context so requires, shall hereinafter be referred to collectively as "**Parties**" and individually as "**Party**").

#### **RECITALS:**

- A. The Technology Owner is Universiti Malaysia Pahang.
- B. The Partnership was set up pursuant to the Symbiosis Programme, a Graduates Entrepreneurship Programme initiated by Malaysian Technology Development Corporation ("MTDC") in its effort to promote the commercialisation of public-funded research and development from public universities, research institutions and polytechnics.
- C. The Technology Owner through its research and development arm including the Main Inventor as set out in **Schedule I** has discovered, created, invented and/or developed the Product (defined below) and the Technology (defined below).
- D. The Partnership wishes to obtain and the Technology Owner wishes to grant the Partnership the right to carry out Technology and Market Validation (defined below) of the Product as set out in further detail below in order to determine the viability of the Product for Commercialisation (defined below) on the terms and conditions set out herein.
- E. The Partnership also wishes to obtain and the Technology Owner agrees to provide to the Partnership Consultancy Services (defined below) and assistance relating to the Product and the Technology in order to allow the Partnership to effectively carry out the Technology and Market Validation on the terms and conditions set out herein.

NOW, THEREFORE, in consideration of the mutual understanding, promises and covenants contained herein and other good and valuable consideration, the receipt and sufficiency of which are hereby acknowledged, the Parties, intending to be legally bound, do hereby agree as follows:-

1

## **DEFINITION & INTERPRETATION**

1.1 For the purposes of this Agreement, the following words and phrases shall, where they appear in capital letters, have the following meaning:-

Approvals and Licenses	means relevant trial approvals (if any), certificates, approvals, licenses, no-objection certificates, sanctions, permits, permissions, waivers and consents from the relevant authorities as required under all applicable laws or reasonably recommended by the Technology Owner's counsel to enable the Partnership to use the Technology and Commercialise the Product;
Commercialisation	means the conduct of relevant trials (if any); manufacture and production at a commercial scale sufficient to meet and supply the demands of consumers; the use; the marketing, promotion, distribution, offer for sale, sale and dealing in the course of trade; the provision of after sales services including response to inquiries and complaints of consumers, relevant service providers and authorities; the appointment of licensees and sub licensees for the purpose of carrying out any of the foregoing activities and the word "Commercialise" or other grammatical variations thereof shall have the same meaning;
<b>Consultancy Services</b>	means the services to be provided by the Technology Owner to the Partnership in relation to the Technology and Market Validation as set out in this Agreement including as more specifically set out in Schedule III herein;
Effective Date	means the date of this Agreement;
Exclusive	means sole and exclusive to the exclusion of all third parties including the person granting the right;
Facility	means the place, facility or factory where the Product is manufactured;
Fee	means the fee payable by the Partnership to the Technology Owner pursuant to Clause 8.1 below;

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means any cause affecting the performance of this **Force Majeure** Agreement arising from or attributable to acts, events, omissions or accidents beyond the reasonable control of the Party to perform and, without limiting the generality thereof, includes:-(1)strikes, lockouts or other industrial actions; (2)civil commotion, riot, invasion, war threat or preparation for war; fire, explosion, storm, flood, earthquake, (3)subsidence, epidemic or other natural physical disaster; (4) impossibility of the use of railways, shipping, aircraft, motor transport or other means of public or private transport; (5) change in law affecting any Party's obligations under this Agreement; or (6) political or governmental interference or change of government policy or actions materially affecting the operations and obligations of a Party under this Agreement; means the funds received from MTDC under **Grant Monies** Commercialisation of Research & Development Fund (CRDF) and disbursed to the Partnership to enable the Partnership to carry out its obligations under this Agreement; means all improvements, modifications or adaptations Improvements to the Product or the Technology or any part thereof during the Term of this Agreement; means:-**Intellectual Property** (1)all rights, title and interest in inventions; manner, method or process of manufacture; method or principle of construction; all plans, drawing or design; scientific, technical or engineering information or document;

6

- (2) all rights, title and interest in Improvements, modification or development of any of the foregoing;
- (3) patent, application for a patent, right to apply for a patent or similar rights;
- (4) trade secret, know-how, confidential information or right of secrecy or confidentiality;
- (5) copyright or other rights in the nature of copyright subsisting in any works or other subject matter;
- (6) any registered or unregistered trade or service marks including all goodwill therein;
- (7) all industrial, proprietary and intellectual property rights, title and interest whether conferred by statute, at common law or in equity whether existing now or in the future including all intellectual property rights which fall within the definition of intellectual property rights contained in Article 2 of the World Intellectual Property Organisation Convention of July 1967; and
- (8) any other rights, title or interest arising from intellectual activities in the scientific, literary or artistic fields;

whether vested before or after the date of this Agreement and whether existing in Malaysia or otherwise in any part of the world, whether or not registered or registrable and for the duration of the rights;

means the individual(s) with the particulars as set out in **Schedule I** herein, who is/are responsible for the discovery, creation, invention and/or development of the Product and Technology;

**Main Inventor** 

Marks

Milestones on Technology Readiness

Process

Product

**Product Literature** 

Technology

Technology and Market Validation

Term

refers to the registered and unregistered trade or service marks in respect of the Products as listed out under **Part E of Schedule II** herein;

refers to the milestones to be achieved by each party as set out in **Schedule III** herein;

means the process of manufacture or production of the Product, including all the particulars described in Part B of Schedule II herein, as well as particulars of all apparatuses, machines, equipment techniques, and methods for production and manufacture of the Product on an industrial or commercial scale, and any Improvements to any of the foregoing;

means the article or substance in the form and with the specifications as set out in **Part A of Schedule II** herein;

means documents setting out information relating to the Product including the benefits of using the Product, instructions on method of use, safety precautions etc., to be distributed to members of the public and potential customers;

means the Process, the ingredients or components, formulations, specification and composition of the Product; and all technical knowledge or data including engineering data (if any), specifications and materials, know-how, procedures, process flows, design, drawings, specifications and other information and materials, regardless of form (whether written documents or unwritten know how) related to the Product or the Process as set out in **Part C of Schedule II** herein;

means the actions to be carried out by the Partnership in relation to the Product including all such activities to determine if the Product is technically and commercially viable for Commercialisation as more specifically set out in Schedule III;

means the period during which the Agreement continues to be in force pursuant to **Clause 5**; and

**Undertaking** means an agreement to be signed between the Partnership, the Technology Owner and the Main Inventor which shall set out the commitments and obligations of the Technology Owner and the Main Inventor in relation to this Agreement as well as representations and warranties of the Technology Owner and the Main Inventor in relation to the Product and Technology.

1.2

In this Agreement, unless there is something in the subject or context inconsistent with such construction or unless it is otherwise expressly provided:-

- 1.2.1 words denoting one gender include all other genders and words denoting the singular include the plural and vice versa;
- 1.2.2 words denoting persons include corporations, and vice versa, and also include their respective heirs, personal representatives, successors in title or permitted assigns, as the case may be;
- 1.2.3 where a word or phrase is given a defined meaning in this Agreement, any other of speech or other grammatical form in respect of such word or phrase has a corresponding meaning;
- 1.2.4 where a word or phrase indicates an exception to any of the provisions of this Agreement, and a wider construction is possible, such word or phrase is not to be construed *ejusdem generis* with any foregoing words or phrases and where a word or phrase serves only to illustrate or emphasis any of the provisions of this Agreement such word or phrase is not to be construed, or to take effect, as limiting the generality of such provision;
- 1.2.5 any reference to a recital, sub-paragraph, paragraph, sub-clause, clause, schedule, appendix, annex, or party, is to the relevant recital, sub-paragraph, paragraph, sub-clause, clause, schedule, appendix, annex or party of, or to, this Agreement and any reference to this Agreement or any of the provisions hereof includes all amendments and modifications made to this Agreement from time to time in force;
- 1.2.6 any reference to a statutory provision includes any modification, consolidation or reenactment thereof for the time being in force, and all statutory instruments or orders made pursuant thereto;
- 1.2.7 any reference to "pay" or cognate expressions, includes payments made in cash or by way of bank drafts (drawn on a bank licensed to carry on banking business under the provisions of the Financial Services Act 2013) or effected through inter-bank transfers to the transferable, cleared

funds and the word "Ringgit" and the abbreviation "RM" mean the lawful currency of Malaysia;

- 1.2.8 any reference to "writing", or cognate expressions, includes, any communication effected by facsimile transmission, email or other instantaneous means;
- 1.2.9 any reference to a "business day" is to a day (not being a Saturday or Sunday) on which banks, licensed to carry on banking business under the provisions of the Financial Services Act 2013, are open for business in Kuala Lumpur and any reference to a "day", "week", "month" or "year" is to that day, week, month or year in accordance with the Gregorian calendar; and
- 1.2.10 if any period of time is specified from a given day, or the day of a given act or event, it is to be calculated of that day and if any period of time falls on a day, which is not a business day, then that period is to be deemed to only expire on the next business day.
- 1.3 The Recitals and Schedules to this Agreement shall have effect and be construed as an integral part of this Agreement, but in the event of any conflict or discrepancy shall, for the purposes of this interpretation and enforcement of this Agreement, be resolved by:-
  - 1.3.1 giving the provisions contained in the clauses of this Agreement, priority and precedence over the provisions contained in the Schedules and Recitals to this Agreement; and
  - 1.3.2 giving the provisions contained in the Schedules of this Agreement, priority and precedence over the provisions contained in the Recitals to this Agreement.
- 1.4 The table of contents, headings and sub-headings in this Agreement are inserted merely for convenience of reference and shall be ignored in the interpretation and construction of any of the provision herein contained.

#### 2 CONDITION PRECEDENT

2.1 It is a condition precedent to the coming into effect of this Agreement that the Technology Owner shall execute and shall procure that the Main Inventor executes the Undertaking in the form annexed herewith as Schedule IV ("Condition **Precedent**").

- 2.2 The Condition Precedent must be satisfied within fourteen (14) days from the date of this Agreement or such extended time as mutually agreed upon by the Parties in writing.
- 2.3 In the event that the Technology Owner fails to execute or procure the execution by the Main Inventor of the Undertaking, this Agreement shall not come into effect and shall be deemed void ab-initio with no obligations or liability on the part of either Party.

#### **3 GRANT OF RIGHTS**

4

- 3.1 The Technology Owner hereby grants to the Partnership, and the Partnership hereby accepts the Exclusive, royalty free, worldwide right to carry out the Technology and Market Validation including but not limited to:-
  - (i) testing the Product either by the Partnership themselves or through a third party laboratory or research institution;
  - (ii) conducting market surveys and tests to determine if there is a market for the Product;
  - (iii) promotion and marketing of the Product to relevant members of the public through any form of media and publication including holding of seminars, workshops, publication of articles in printed media, meeting and disclosing details of the Product with professionals in the relevant industry and members of the public, distribution of brochures, flyers and other promotional material;
  - (iv) producing the Product Literature in relation to the Product for the above purpose or using such Product Literature as provided by the Technology Owner as the case may be; and
  - (v) subject to written notification to the Technology Owner, appointing third parties and sub-contractors for any of the above purposes.
- 3.2 For the avoidance of any doubt, the Technology Owner shall not during the Term of this Agreement use or disclose to any third party the Technology or the Product in any form or manner including for any form of testing of the Technology or the market for the Product, Commercialisation, licensing or other form of collaboration with any third party, market survey or research, without consent in writing from the Partnership.

# **RESPONSIBILITIES OF THE TECHNOLOGY OWNER AND THE PARTNERSHIP**

4.1 Both Parties shall carry out their obligations as set out in Schedule III within the timelines stipulated therein.

4.2 The Technology Owner shall be responsible for and shall ensure that it carries out and procures that the Main Inventor carries out in a timely and skilful manner the Consultancy Services as set out in Schedule III. The Technology Owner shall execute and procure that the Main Inventor execute the Undertaking in accordance to Sub-Clauses 2.1 and 2.2 of this Agreement. The Technology Owner shall comply and shall ensure compliance by the Main Inventor with the terms and conditions of this Agreement and the Undertaking throughout the Term.

- 4.3 The Consultancy Services shall include the following as more particularly set out in Schedule III:-
  - 4.3.1 providing the Partnership with a sufficient quantity of the Product to carry out the Technology and Market Validation as requested by the Partnership from time to time;
  - 4.3.2 carrying out all necessary quality control procedures for the Product and the manufacture and production thereof at the Facility to ensure that it complies with the product specifications and is of the highest quality and safe for use;
  - 4.3.3 providing the Partnership the Product Literature and all relevant information to conduct the Technology and Market Validation for the Product, including answering any queries that may be raised by consumers, health care providers and relevant authorities relating to the Product and the Technology;
  - 4.3.4 providing continuous technical assistance or advice and cooperating with the Partnership whenever required for the purposes of carrying out the Technology and Market Validation of the Product;
  - 4.3.5 making the required modifications or Improvements to the Technology and the Product including such modifications or Improvements requested by the Partnership and modifications or Improvements to ensure that the Product is ready for Commercialisation, and obtaining the requisite Approvals and Licenses (if any); and
  - 4.3.6 obtaining and procuring the required Approvals and Licenses (if any) to conduct relevant trials (if any) for the Product and thereafter to use and implement the Technology and Commercialise the Product and carry out all activities related to the Technology and Market Validation as set out in Schedule III.

- 4.4 In the event that the Main Inventor leaves his employment with the Technology Owner, the Technology Owner shall immediately notify the Partnership and shall make the necessary arrangement for an individual/ individual(s) with similar expertise and competency level to continue to provide assistance and support to the Partnership and shall ensure that all the obligations and liabilities of the Main Inventor under this Agreement and the Undertaking shall apply to such individual for the remaining Term of this Agreement and the Undertaking.
- 4.5 For the avoidance of any doubt, the Technology Owner shall ensure that there is continued assistance and support to the Partnership as envisaged under this Agreement at all times notwithstanding the absence of the Main Inventor or inability of the Main Inventor to provide the assistance whether due to the Main Inventor ceasing employment with the Technology Owner, the Main Inventor's work schedule and other commitments, the Main Inventor being on vacation, leave, medical or maternity leave or sabbatical, due to death, injury or incapacitation of the Main Inventor or for any other reason.
- 4.6 The Partnership shall provide the Technology Owner with any data, materials, documentation or any other form of information obtained by the Partnership through the Technology and Market Validation activities carried out by the Partnership, and where the Technology Owner has access to any of the foregoing, it shall not be disclosed to any third party or used by the Technology Owner in any form or manner whatsoever except as otherwise provided under this Agreement.
- 4.7 The Technology Owner shall comply and shall procure that the Main Inventor complies with the terms of this Agreement and any non-disclosure agreement (if applicable) entered into with the Partnership.
- **4.8** The Technology Owner will take all necessary action to enforce the Main Inventor's obligations under and pursuant to this Agreement.
- 4.9 The Technology Owner shall ensure that independent of the Main Inventor, it has all the necessary information including Confidential Information relating to the Technology and the Product in order to be fully competent and able to continue to provide the necessary assistance and support to the Partnership throughout the Term of this Agreement.

#### 5 TERM AND EXTENSION OF TERM

- 5.1 This Agreement shall, irrespective of the diverse dates upon which the Parties may have signed this Agreement, come into force on the Effective Date.
- 5.2 This Agreement shall continue and remain in full force and effect for a period of twelve (12) months from the Effective Date (the "**Term**") or until such time as:-

- (i) either party terminates this Agreement pursuant to the terms of this Agreement; or
- (ii) the Partnership decides that it is ready to proceed with licensing of the Technology and Commercialisation of the Product and exercises the right to a license as set out in **Clause 6** below.
- 5.3 The Partnership shall have the right to extend the Term of this Agreement for a further term as required by the Partnership, if it is deemed necessary by the Partnership, for the purposes of carrying out Technology and Market Validation of the Product, without any additional consideration or fee payable to the Technology Owner.

#### **6 RIGHT TO OBTAIN LICENCE**

- 6.1 At any time during the Term or within six (6) months of expiry of the Term or any extension of the Term, the Partnership shall have the sole and Exclusive right to obtain from the Technology Owner a license to use and Commercialise the Technology and the Product upon such terms and conditions to be agreed between the Parties.
- 6.2 The Technology Owner shall license the Technology to the Partnership at a reasonable consideration and for a term to be agreed upon between the Parties. The Parties shall negotiate the terms and conditions of the license agreement in good faith.

#### 7 **IMPROVEMENTS**

7.1 If at any time during the Term the Technology Owner makes, discovers, devises, invents or otherwise acquires any Improvements to the Product or the Technology whether or not pursuant to a request by the Partnership under Sub-Clause 4.3.5 above, the Technology Owner shall immediately notify the Partnership and shall if so requested by the Partnership obtain the manufacture of and supply to the Partnership a sufficient quantity of the Product incorporating such Improvements and grant the Partnership full rights to continue the Technology and Market Validation on the Product incorporating such Improvements without any additional fees or payment. All Intellectual Property rights in such Improvements by the Technology Owner shall belong to the Technology Owner subject to the foregoing right to use granted to the Partnership under this Agreement.

#### FINANCIAL PROVISIONS

8

#### 8.1 Payment of Consultancy Fee

- 8.1.1 In consideration of the Technology Owner carrying out the Consultancy Services in accordance with the Milestones on Technology Readiness, the Partnership shall pay to the Technology Owner the prescribed fee as set out in Schedule I ("Consultancy Fee"), payable upon achievement of the Milestones on Technology Readiness in such amounts and at such times as stipulated in Schedule III.
- 8.1.2 First payment of the Consultancy Fee shall be payable upon three (3) months of disbursement of Grant Monies to the Partnership subject to compliance by the Technology Owner with the Milestones on Technology Readiness set out in **Schedule III**. No Consultancy Fee or other payment shall be due or payable by the Partnership to the Technology Owner prior to the disbursement of the Grant Monies to the Partnership.

8.1.3 For the avoidance of doubt, the Consultancy Fee is deemed inclusive of out-of-pocket (such and shall cover all expenses as transportation/travelling costs, accommodation, meals and other expenses including subsistence allowances) incurred by the Main Inventor performing their obligations under this Agreement, and no separate claim shall be made for the same. No separate fee or disbursements shall be payable by the Partnership to the Main Inventor at any time.

#### 9 CONFIDENTIALITY

- 9.1 Each Party shall maintain secret and confidential all information relating to the Technology and the Product regardless of form and regardless in the case of the Technology Owner whether such information originated from itself or the Main Inventor for the duration of this Agreement. Each Party shall not use such information or disclose the same to any third party, save only for the purpose of carrying out each Party's obligations under and pursuant to this Agreement and may only disclose to those of its employees, agents, contractors and third parties to whom and to the extent that such disclosure is reasonably necessary for the above purpose.
- 9.2 Each Party shall procures that all of its agents, employees or contractors who have or are given access to any information relating to the Product or the Technology for the purposes set out in **Sub-Clause 9.1** shall sign a non-disclosure agreement agreeing to keep all information provided secret and confidential at all times and not to disclose the same to any third party or use it for any purpose other than as authorised. Both

Parties acknowledge and agree that it will not be possible for the Partnership to obtain a non-disclosure agreement or any confidentiality undertaking from all third parties to whom the Partnership seeks to promote and sell the Product or obtain feedback from in the course of conducting the Technology and Marketing Validation and that the Partnership shall use its discretion to determine when such a non-disclosure agreement or confidentiality undertaking is required. The Partnership shall not be responsible for any unauthorised use or disclosure of information relating to the Product or the Technology by any such third party.

- **9.3** The Technology Owner shall maintain secret and keep confidential all or any data, information, materials or documentation obtained through the Technology and Market Validation for the Product carried out by the Partnership or by reason of this Agreement, or any information relating to the activities and business of the Partnership where the Technology Owner has access to the same.
- 9.4 Neither Party shall disclose any terms or conditions of this Agreement to any third party without the prior written consent of the other Party. However, a Party may disclose the terms and conditions of this Agreement:-
  - 9.4.1 on a need-to-know basis to its legal and financial advisors/counsels to the extent such Party's activities are expressly permitted by this Agreement and are ordinary and customary business operations; and
  - 9.4.2 to a third party in connection with (i) an equity investment in such Party, transaction by such Party, or (ii) a merger, consolidation, change in control of similar transaction by such Party, or (iii) the transfer or sale of all or substantially all of the assets of such Party.
- 9.5 The obligations of this **Clause 9** shall not apply to the extent that a Party is required to disclose information by applicable law, regulation or bona fide legal process, provided that the Party required to make the disclosure takes reasonable steps to restrict and maintain confidentiality of such disclosure and provides reasonable prior notice to the other Party.

#### **10** NON-COMPETITION

The Technology Owner agrees that during the Term of this Agreement and for six (6) months thereafter, the Technology Owner will not, directly or indirectly, engage in any same or similar arrangement, collaboration, cooperation or business in relation to the Technology or the Product with any third party without the prior written approval from the Partnership. The Technology Owner will also not seek to Commercialise the Product or the Technology or promote, market, use, produce, manufacture, offer to sell, sell, distribute or otherwise deal with the Product or the Technology or attempt or seek to do any of the foregoing in any way or manner either by itself or in collaboration with any third party during the Term of this Agreement.

#### 11 TERMINATION

- 11.1 Either Party shall be entitled to issue a notice to the other Party terminating this Agreement if, at any time:-
  - 11.1.1 the other Party commits any continuing or material breach of any of its obligations under this Agreement which either:-
    - (a) is incapable of remedy; or
    - (b) if capable or remedy, is not remedied within thirty (30) days of the Party which is not in breach giving the defaulting party written notice of the breach and to remedy the breach; or
  - 11.1.2 a petition is presented or an order is made or a resolution is passed for the winding up of the other Party; or
  - 11.1.3 an administrator or receiver or receiver and manager is appointed over, or distress, attachment or execution is levied or enforced upon, any part of the assets or undertaking of the other Party; or
  - 11.1.4 the other Party becomes insolvent or is unable to pay its debts or admits in writing its inability to pay its debts as they fall due or enters into any composition or arrangement with its creditors or makes a general assignment for the benefit of its creditors; or
  - 11.1.5 the other Party ceases or threatens to cease to carry on the whole or any substantial part of its business (except for the purposes of a bona fide reconstruction or amalgamation which would not result or cause any failure or inability to duly perform or fulfill any obligation under this Agreement).
- 11.2 The Partnership shall be entitled at any time during the Term of this Agreement to terminate this Agreement without providing any reason and without thereby incurring any liability, by giving the Technology Owner written notice of such termination.
- 11.3 Following the giving of a notice terminating this Agreement and pursuant to any of the provisions of this Agreement, neither Parties shall have any further rights or obligations under this Agreement to the other Party, except in respect of:-
  - 11.3.1 the confidentiality obligations except where such obligations are stated to be limited to the Term of this Agreement;

- 11.3.2 any right or obligation under this Agreement which is expressed to apply or continue to be binding after the termination of this Agreement, or which by its nature is intended to continue after termination of this Agreement;
- 11.3.3 any rights or obligations which have accrued to either Party prior to such termination including any rights in respect of any breach of any of the provisions of this Agreement by; and
- 11.3.4 any representations, warranties and indemnities provided in this Agreement.
- 11.4 Upon expiration or termination of this Agreement for any reason:-
  - 11.4.1 The Partnership shall cease to carry out any activities including any form of technology or market validation in relation to the Product, either directly or indirectly, and the Partnership shall as soon as practicable return to the Technology Owner the sample products supplied by the Technology Owner, any Product Literature and other materials relating to the Product or the Technology received from the Technology Owner; and
  - 11.4.2 The Technology Owner shall return to the Partnership any data or materials containing information related to the Technology and Market Validation activities carried out by the Partnership and shall not use such information, data or material for any purpose whatsoever including Commercialisation of the Product or seeking collaboration with any third party.

#### 12 REPRESENTATION AND WARRANTIES

- 12.1 The Technology Owner represents and warrants to the Partnership that:-
  - 12.1.1 it has all the necessary authority to enter into this Agreement;
  - 12.1.2 the execution by the Technology Owner of, and the performance of its obligations under this Agreement require no governmental or other approvals whatsoever and if so required, all such approvals have been obtained;
  - 12.1.3 it has executed or procured to be executed all such documents and done or procured to be done all such acts and things as may be necessary to give full effect to all provisions of this Agreement;

- 12.1.4 it has not, nor at any time will it be, a party to any contract or other arrangement of any nature that will materially interfere with its full, due and complete performance of this Agreement;
- 12.1.5 neither the execution nor the performance of any of the matters contemplated in this Agreement:
  - (a) contravene or constitute a default under any provision contained in any agreement, instrument, law, judgment, order, licence, permit or consent by which the Technology Owner or any of its assets are bound or affected; or
  - (b) cause any limitation on the Technology Owner or the powers of its directors whether imposed by or contained in its charter, constitution or in any other law, order, judgment, agreement, instrument or otherwise, to be exceeded;
- 12.1.6 it is free from legal encumbrances and no litigation, arbitration or administrative proceeding or claim which might by itself or together with any other such proceedings or claims, is presently in progress or pending or, to the best of the knowledge, information and belief of the Technology Owner, threatened or foreseeably contemplated against the Technology Owner which either:
  - (a) would or might materially and adversely affect the exercise or performance of any of the rights and/or obligations of the Technology Owner under this Agreement;
  - (b) by itself or together with any such proceedings or claims would or might materially and adversely affect the financial condition, business or operations of the Technology Owner;
  - (c) would or might materially and adversely affect the validity or enforceability of this Agreement; or
  - (d) would be prejudicial to the Partnership's interests;
- 12.1.7 it shall maintain the registration and pay all fees (where applicable) for any licenses, patent, trademarks and other approval in relation to the Product and the Technology during the Term of this Agreement; and
- 12.1.8 the Technology Owner and the Main Inventor have the expertise, experience and resources required to perform their obligations under this Agreement and shall perform the same with due care and skill, recognising that the Partnership is relying on the provision by the

Technology Owner and the Main Inventor of such expertise to carry out the Technology and Market Validation of the Product.

#### **13 INTELLECTUAL PROPERTIES PROTECTION AND PROCEDURES**

#### 13.1 **Protection Costs**

The Technology Owner hereby agrees to bear in full the prescribed fees chargeable for renewing the Intellectual Property rights protection for the Product and the Technology for the duration of the Term.

#### 13.2 INTELLECTUAL PROPERTY Representation and Warranties

In addition to any other warranty expressed or implied in the Agreement or by statute, the Technology Owner hereby represents and warrants to the Partnership that:-

- 13.2.1 except where Sub-Clause 13.2.2 applies, the Technology and the Product were discovered, created, invented or developed by the Main Inventor while under the employment of the Technology Owner, and the Technology Owner is the exclusive legal and beneficial owner of all proprietary rights including Intellectual Property rights in the Technology and the Product free of any liens charges, claims or encumbrances whatsoever and thereby has full legal right and authority to provide the Partnership with the rights granted under this Agreement and to perform its obligations under this Agreement;
- 13.2.2 where the Intellectual Property rights in the Technology and the Product belong to a third party, the Technology Owner is duly licensed and authorised by such third party to grant the Partnership the rights granted under this Agreement and to perform its obligations under this Agreement;
- 13.2.3 the Technology Owner has carried out all relevant freedom to use searches around the world and the Technology and Market Validation activities and the use of the Technology and the use, production, manufacture marketing, promotion, sale, distribution, import, offer for sale and sale of the Product and the use and possession of all data, information, materials or documentation, provided by the Technology Owner to the Partnership do not and shall not infringe any proprietary rights including Intellectual Property rights of any third party;
- 13.2.4 as of the date of this Agreement, neither the Technology Owner or any other party has received any notices, claims or demands of legal proceedings relating to the use, production, manufacture marketing,

promotion, sale, distribution, import, offer for sale or sale of the Product or the use of the Technology or any element thereof, and there are no third party rights or claims which would restrict the exploitation and/or Commercialisation of the Product or the Technology;

- 13.2.5 there has been no attempt or action taken by the Technology Owner or any third party either alone or in collaboration at any time in the past to Commercialise the Product and neither the Technology Owner nor the Main has at any time in the past granted a license to the Technology to any third party or to conduct any form of testing or market survey or research relating to the Product;
- 13.2.6 to the best of the Technology Owner's knowledge there is no infringement or unauthorised use of the Technology or the Product by any third party; and
- 13.2.7 the Technology is sufficient to produce and manufacture the Product on an industrial scale and for Commercialisation of the Product.

#### 13.3 Infringement

13.3.1 In the event either the Technology Owner or the Partnership becomes aware of any infringement of the Intellectual Property rights in the Technology or the Product ("IPR"), the Parties agree to confer to decide whether to enforce such IPR, and if enforcement is to be undertaken, whether it is to be at the sole expense and management of one of them, or jointly by both Parties upon mutually satisfactory costs arrangements.

- 13.3.2 In the event the Partnership, elects to initiate litigation to enforce such IPR, the Parties hereby agree to co-operate with one another in such litigation and each Party will provide the other with all pertinent information in its possession which may be helpful in such litigation. The Partnership shall have the right to dispose of such action(s), in whatever reasonable manner it determines to be in the best interests of this Agreement.
- 13.3.3 The Technology Owner may at any time initiate or defend litigation at its own cost and expense, in relation to such IPR, and shall ensure that the Partnership is notified of the same, and kept updated on the progress and outcome of such litigation, at all times.

#### 13.4 Indemnity

13.4.1 Without prejudice to any other rights and remedies of the Partnership under this Agreement or applicable law, the Technology Owner agrees

21

to indemnify, defend and hold harmless the Partnership, its successors and assigns, and its and their officers, directors, employees, sublicensees and agent (the "**Indemnified Party**"), against any and all claim, action, damage, loss, liability, cost, charge, expense, outgoing or payment, (including attorney's fees and court costs on an indemnity basis) which the Indemnified Party may pay, suffer, incur or be liable for which arise directly or indirectly our of or in connection with:-

- (i) any breach of the representations and warranties in Clause 12 or Sub-Clause 13.2 or any of the representations or warranties being false, inaccurate or misleading;
- (ii) the Product or Technology infringing any proprietary rights including Intellectual Property rights of any third party or any demand, claim, threat, legal proceedings by any third party that the use, production, manufacture marketing, promotion, sale, distribution, import, offer for sale and sale of the Product or the Technology infringes any proprietary rights including Intellectual Property rights of such third party;
- (iii) the Technology being unable or insufficient to produce or manufacture the Product on an industrial scale or to Commercialise the Product;
- (iv) the Product being unsafe for use, defective or not being of merchantable quality, fit for its purpose or in compliance with any description of the Product or any implied warranty under applicable law including any demand, claim, threat, legal proceedings by any third party of any of the foregoing; and
- (v) any action taken or penalty imposed by the authorities on the Partnership in relation to the possession and use of the Technology or any part thereof or the Commercialisation of the Product.
- 13.4.2 Without prejudice to any other rights and remedies of the Partnership under this Agreement including the indemnity under **Sub-Clause 13.4.1** above or any other applicable law, the Partnership may in its absolute discretion give the Technology Owner the conduct of the defence to any claim or action in respect of any of the foregoing with the right to take back the conduct of the defence if it does not believe the Technology Owner is conducting the same competently. So long as the Technology Owner has conduct of the proceedings, the Partnership shall not admit liability or otherwise attempt to settle the same claim or action subject to the Technology Owner providing to the Partnership's reasonable

satisfaction security for any costs or liabilities the Partnership may incur by reason of the Technology Owner's conduct of such defence.

13.4.3 Without prejudice to any other rights and remedies of the Partnership under this Agreement including the indemnity under Sub-Clause 13.4.1 or applicable law, if the Partnership's use or possession of the Product or Technology is held by a court to constitute an infringement of a third party's intellectual property rights or if the Partnership is advised by legal counsel that such use or possession is likely to constitute such an infringement then the Technology Owner shall promptly, at its own expense and costs and to the best of its abilities:-

- (a) procure for the Partnership the right to continue using the Product or Technology or any portion of the same, as the case may be; or
- (b) modify or replace the Product or Technology at the Technology Owner's sole cost and expense (without any degradation in quality) so as to avoid the infringement (in which event the Technology Owner shall compensate the Partnership for the amount of any direct loss and/or damage sustained or incurred by the Partnership during such modification or replacement); or
- (c) if (a) or (b) cannot be accomplished on reasonable terms, refund all payments and/or fees made by the Partnership pursuant to this Agreement in Clause 8 and compensate the Partnership for the amount of any loss and/or damage sustained or incurred by the Partnership by reason of loss of use of the Product or Technology.

#### 14 COSTS

- 14.1 Subject to Sub-Clause 14.2, each Party shall bear its own solicitors' costs of, and incidental to, the preparation of this Agreement.
- 14.2 The Technology Owner shall be responsible for the stamping of this Agreement and all costs and expenses incurred in connection thereof.

#### 15 COMMUNICATIONS

#### 15.1 Notices

Any notice or other communication to be given under or in respect of this Agreement shall be in writing and may be delivered, given or sent by:

15.1.1 hand;
15.1.2 registered post or express post or courier or other fast postal service; or
15.1.3 facsimile transmission or other instantaneous electronic media including electronic mail.

#### 15.2 Address

Any notice or other communication to be given under or in respect of this Agreement shall be delivered, given or sent to the addressee at the address or facsimile transmission number, and marked for the attention of the person or officer of the addressee, set out below, or at such other address or facsimile transmission number or marked for the attention of such other person or officer person as the addressee may give notice of to the other Party from time to time in accordance with this Agreement:

#### For the Technology Owner

Address	
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: LEVEL 2, CANSELERI TUN ABDUL RAZAK, UNIVERSITI MALAYSIA PAHANG, 26600 PEKAN, PAHANG DARUL MAKMUR

Tel : 09-424 5004 / 5410

Fax : 09-424 5444

Email address : mashitah@ump.edu.my and nizamrashid@ump.edu.my

Attention of : DEPUTY VICE CHANCELLOR (RESEARCH AND INNOVATION) EN. MOHD NIZAM BIN ABDUL RASHID (TECHNOLOGY TRANSFER OFFICE)

#### For the Partnership

Address : [Symbiosis Hub, Block B Level 1, UPM-MTDC Technology Centre III, Universiti Putra Malaysia, 43400, Serdang, Selangor.]

Fax No. : [N/A]

Email address : [orcagreentech@gmail.com]

Attention of : [En. Arif Bin Md Zain, (Chief Executive Officer)]

#### 15.3 Language

Any notices and communications to be given under or in respect of this Agreement shall be in the English language or, if in any other language, accompanied by a translation thereof in the English language, certified to be a true and correct translation of the original.

#### 15.4 Time of service

Any notices and communications to be given under or in respect of this Agreement shall be deemed to have been duly served upon and received by the addressee:-

- 15.4.1 if delivered by hand prior to 5.00p.m. on a business day, at the time of delivery or, if delivered by hand at any other time, at 9.00a.m. on the next business day following the date of such delivery;
- 15.4.2 if sent by registered post, first class post or express or air mail or other fast postal service, on the 3<sup>rd</sup> business day falling after the date of dispatch; or
- 15.4.3 if transmitted by facsimile or electronic mail upon transmission if transmission is on a business day prior to 5.00p.m., otherwise at 9.00a.m. on the following business day.

#### 15.5 **Proof of service**

In proving the giving of a notice or any other document under or in respect of this Agreement, it shall be sufficient to show:

- 15.5.1 in the case of facsimile transmission or other instantaneous electronic media, that the notice or other document was duly sent; and
- 15.5.2 transmitted from the dispatching terminal as evidenced by a transmission report generated by the dispatching terminal.

#### 16 DISPUTE RESOLUTION COMMITTEE

#### 16.1 **Composition**

Any dispute between the Parties in respect of any matter arising under this Agreement may be referred by either Party to a dispute resolution committee (the **"Dispute Resolution Committee**") which shall comprise:

- (a) one (1) independent representative mutually agreed by the Technology Owner <u>AND</u> the Partnership;
- (b) two (2) representatives to be appointed by the Technology Owner; and
- (c) two (2) representatives to be appointed by the Partnership.

#### 16.2 Independent Expert

The Dispute Resolution Committee may appoint an independent expert to advise it on any matter referred to it and all costs, fees and expenses of any independent expert so appointed shall be borne equally by the Parties.

#### 16.3 Amicable Settlement

The Dispute Resolution Committee shall determine its own procedures. The Dispute Resolution Committee shall meet and endeavour to achieve an amicable settlement between the Parties in respect of any dispute referred to it. If any matter, dispute or claim which is referred to the Dispute Resolution Committee cannot be amicably settled by both Parties within thirty (30) days after the date of referral, either Party may refer the matter, dispute or claim to the Courts of Malaysia.

#### 16.4 **Procedures**

Each member of the Dispute Resolution Committee shall have one (1) vote each.

#### 16.5 Binding

Any decisions of the Dispute Resolution Committee shall be binding on the Parties.

#### **17 NATURE OF AGREEMENT**

#### 17.1 Entire Agreement

This Agreement:

17.1.1 constitutes the entire agreement and understanding between the Parties with respect to the matters dealt with in this Agreement; and

17.1.2 was not entered into by the Parties in reliance of any agreement, understanding, warranty or representation of any Party not expressly contained or referred to in this Agreement.

#### 17.2 Counterpart

This Agreement may be executed in any number of counterparts or duplicates each of which shall be an original, but such counterparts or duplicates shall together constitute but one and the same agreement.

#### 17.3 Amendments & additions

No amendment, variation, revocation, cancellation, substitution or waiver of, or addition or supplement, of any of the provisions of this Agreement shall be effective unless it is in writing and signed by both Parties.

#### 17.4 Successors & Assigns

This Agreement shall be binding upon and inure for the benefit of the respective permitted assigns and successors-in-title of the Parties.

#### 17.5 Assignment

Either Party shall not assign or transfer all or any part of their respective rights under this Agreement or delegate their performance under this Agreement without prior written consent of the other Party. Any assignment, transfer or delegation which is made without such prior written approval shall constitute a material breach of this Agreement.

#### 17.6 No Partnership or Agency

The provisions of this Agreement shall not be construed or taken to constitute:

- 17.6.1 a partnership between the Parties;
- 17.6.2 either Party to be the agent of the other Party; or
- 17.6.3 an authority to either Party to represent or bind or pledge the credit of the other Party in anyway.

#### 17.7 General Covenant

Each Party has entered in to this Agreement in good faith and shall give all such assistance and information to the other Party and execute and do and procure all other necessary person or companies, if any, to execute and do all such further acts, deeds, assurance and things as may be reasonably required so that full effect may be given to the terms and conditions of this Agreement.

#### 17.8 Invalidity & Severability

If any provision of this Agreement is or may become ineffective under any written law, or is found by any court or administrative body or competent jurisdiction to be, illegal, void, invalid, prohibited or unenforceable then:

- 17.8.1 such provision shall be ineffective to the extent of such illegality, voidability, invalidity, prohibition or unenforceability;
- 17.8.2 the remaining provisions of this Agreement shall remain in full force and effect; and
- 17.8.3 the Parties shall use their respective best endeavours to negotiate and agree on a substitute provision which is valid and enforceable and achieves to the greatest extent possible the economic, legal and commercial objectives of such illegal, void, invalid, prohibited or unenforceable term, condition, stipulation, provision, covenant or undertaking.

#### 17.9 Force Majeure

17.9.1 The Parties shall not be in breach of their obligations under the Agreement if they are unable to perform or fulfill any of their obligations hereunder as a result of the occurrence of an event of Force Majeure, which causes, or can be reasonably expected to cause, either Party to be unable to fulfill its obligations under this Agreement.

- 17.9.2 If an event of Force Majeure occurs which renders a Party unable to perform its obligations ("Affected Party") under this Agreement or any part thereof, the Affected Party shall:
  - (a) notify the other Party in writing as soon as reasonably practicable of the occurrence of the event of Force Majeure giving full details thereof;
  - (b) take all reasonable measures to mitigate any delay or interruption to the subject matter of this Agreement; and
  - (c) notify the other Party of the cessation of such event of Force Majeure.
- 17.9.3 If an event of Force Majeure continues for a period of more than two (2) months or any one of the Parties reasonably considers such event of Force Majeure to be of such severity that it effectively frustrates the original

intention of this Agreement, then the Parties may by mutual agreement terminate this Agreement.

17.9.4 Notwithstanding any provisions under this **Clause 17.9**, if the occurrence of any event of Force Majeure does not warrant the Parties to terminate this Agreement, the obligations and liabilities of the Parties which are not affected by the event of Force Majeure shall remain in full force and effect.

#### 17.10 Knowledge & Acquiescence

Knowledge or acquiescence by any Party of, or in, any breach of any of the provisions of this Agreement shall not operate as, or be deemed to be, a waiver of such provisions and, notwithstanding such knowledge or acquiescence, such Party shall remain entitled to exercise its rights and remedies under this Agreement, and at law, and to require strict performance of all of the provisions of this Agreement.

#### 17.11 Rights and Remedies

The rights and remedies provided in this Agreement are cumulative, and are not exclusive of any rights or remedies of the Parties provided at law, and no failure or delay in the exercise or the partial exercise of any such right or remedy or the exercise of any other right or remedy shall affect or impair any such right or remedy.

#### 17.12 Governing Law and Jurisdiction

This Agreement shall be governed by the Laws of Malaysia and no suit or other proceedings relating to this Agreement shall be brought or filed in any court other than a court of competent jurisdiction in Malaysia, which shall have exclusive jurisdiction to hear and determine all suits or proceedings arising out of this Agreement.

#### 17.13 Waiver

No failure or delay on the part of either Party hereto to exercise any right or remedy under this Agreement shall be construed or operated as a waiver thereof nor shall any single or partial exercise or any right or remedy, as the case may be. The rights and remedies provided in this Agreement are cumulative and are not exclusive of any rights or remedies provided by law.

### 17.14 General Indemnity

The Parties agree to indemnify and hold each other harmless from any liabilities, claims or demands (including costs, expenses and reasonable solicitor's fees) that may be made by any party for any injuries, death or damage to property to the extent such injury or damage is caused by the negligent acts or omissions or wilful misconduct of one Party or such Party's employees, agents or sub-contractors in the performance of this Agreement.

[The remainder of this page has been intentionally left blank and the next page is execution page]

Technology and Market Validation Agreement

IN WITNESS WHEREOF the Parties hereto have hereunto set their hands on the day and year first abovewritten.

Signed by for and on behalf of [ORCA GREENTECH PLT] LLP No. [0011051-LGN] in the presence of: Name [ARIF BIN MD ZAIN] NRIC No.: [940811065407] Name [AHMAD HAZIQ BIN AHMAD QALAM] NRIC No.: [920618145167] Name of Witness: Sta NUR RABIATUL AD AWITAGH BINTI ASAL AR Signed by for and on behalf of [TECHNOLOGY OWNER] in the presence of: Name: PROFESOR DATO' SRI DR. DAING NASIR ISRAMIM PD.D. CA (M). FCPA (Aus), P. Tech VICE-GRANCELLOR UNIVERSITI MALAYSIA PAHANG Name of Witness: PROFESSOR DATO' DR, MABHITAN MOND, YUSOFF DEPUTY VICE CHANCELLOR (RESEARCH & INNOVATION) UNIVERSITI MALAYSIA PAHANG 26600 PEKAN PAHANG DARUL MAKMUR TEL: 09-424 5004 FAKS: 09-424 5444

Technology and Market Validation Agreement

## SCHEDULE I

Partnership	Name: Orca Greentech PLT				
	Established under the laws of Malaysia.				
	Address: Symbiosis Hub, Block B Level 1, UPM-MTDC				
•	Technology Centre III, Universiti Putra Malaysia, 43400, Serdang,				
	Selangor.				
	LLP No.: 0011051-LGN				
	Name: Universiti Malaysia Pahang				
Technology Owner	Established under: University and University Colleges Act 1971[Act 30]				
	Address: Department of Research and Innovation, Universiti				
	Malaysia Pahang, Pekan 26600 Pahang.				
Main Inventor	Name: Mohd Najib bin Razali				
	NRIC No.: 840809-01-5939				
	Address: Faculty of Chemical and Natural Resources Engineering,				
	Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300				
	Gambang, Pahang				
	Name: Dr. Mohd Aizudin bin Abdul Aziz				
	NRIC No.: 840204-14-6039				
	Address: Faculty of Chemical and Natural Resources Engineering,				
	Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300				
	Gambang, Pahang				
	Name: Prof. Dato' Dr. Rosli bin Mohd Yunus				
	NRIC No.: 631030-10-5235				
	Address: Faculty of Chemical and Natural Resources Engineering,				
	Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300				
	Gambang, Pahang				
	I.				

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	Name: Musfafikri bin Musa				
	NRIC No.: 871115-08-6013				
	Address: Faculty of Chemical and Natural Resources Engineering,				
	Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300				
	Gambang, Pahang				
Consultancy Fee	RM30,000.00 (Excluding tax)				
Other Fees	(only if applicable)				
Payment Method	All payments to be made by the Partnership to the Technology Owner:				
	(a) if by way of cheque, shall be made in the name the Technology				
	Owner or Party representing Technology Owner; or				
	(b) if by way of telegraphic transfer, shall be transferred into the				
	account which will be informed by Technology Owner in				
	writing.				

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Technology and Market Validation Agreement

#### SCHEDULE II

### PART A: DESCRIPTION OF THE PRODUCT

The product relates to grease of produced using a novel thickening agent from the material produced from the patented process.

### PART B: DESCRIPTION OF THE PROCESS

The process is directed to the steps involved in grease formulation using base oil and thickener from industrial waste as well as formulation of the final product i.e. grease itself.

## PART C: DESCRIPTION OF THE TECHNOLOGY

The technology

Technology and Market Validation Agreement

	Particulars	
1.	[Patent Filing No. / Grant No.]	PI 2017400003
2.	Title of Technology	A Thickening Agent for Grease Formulation and Associated Grease Formulation Using The Same
3.	Inventor	<ol> <li>Mohd Najib bin Razali;</li> <li>Dr. Mohd Aizudin bin Abdul Aziz;</li> <li>Prof. Dato' Dr. Rosli bin Mohd Yunus;</li> <li>Musfafikri bin Musa.</li> </ol>
4.	Address	Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, 26300 Gambang Pahang.
5.	Owner/Applicant	Universiti Malaysia Pahang
6.	Address	Department of Research and Innovation, Universiti Malaysia Pahang, 26600 Pekan Pekan Pahang
7.	[Filing Date / Date of Grant]	22 <sup>nd</sup> February 2017

## PART D: PARTICULARS OF THE PATENT

## PART E: TRADE MARKS

Not Applicable

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## SCHEDULE III

Ňo.	Obligations	Responsible Party	Timeline	Deliverables	Payment
•	Education on Product Detail and Presentation Assistance				
	To provide Orca GreenTech PLT the product literature and all relevant information to conduct technology and marketing validation activity. UMP will provide information in form of reading material via email or post to Orca GreenTech PLT. The source of the literature must be reliable and proven	Technology Owner and the Partnership	Month 1 - 3	<ul> <li>i. Receival of notes, reports, related documents and articles; and</li> <li>ii. List of related Standard Operation Procedures.</li> </ul>	15%
	facts. UMP to provide explanation in detail about the product/technologies by discussion. Discussion will be held from time to time or once related problem is faced and can be done by meeting or through phone call. UMP will train members of Orca GreenTech PLT to understand about the product/technologies and able to explain about it to educate people whom are not familiar with the product. UMP also will answer questions enquired by Orca GreenTech regarding the product.	MP	Month 1 - 3	<ul> <li>i. Discussion/meeting request by Orca Greentech PLT;</li> <li>ii. Minutes of meetings;</li> <li>iii. Slides, flow chart and diagram (if any)</li> </ul>	15%

## MILESTONES ON TECHNOLOGY READINESS

2.	Product Testing Assistance						
	Technology Owner to guide The Partnership in types of product testing and analysis required for grease and suggests where to conduct the test. Technology Owner will provide lists of parameters for	Technology Owner	Month 3 -5	i. ii.	Discussion/meeting request by The Partnership; Types of test required;	15%	
	each testing. Technology Owner will assist through the testing procedure and to pass all the parameters for the following tests:- a. Physical Testing (ASTM); and b. Chemical Testing			iii. iv.	List of parameters for each tests; Testing procedures.		
	Technology Owner will discuss with The Partnership on the feedback received from the testing and ways to improve the product.						·
	Product Supply and Improvement Support						
	Technology Owner to supply The Partnership. Products that have been manufactured and the quality is tested according to SOP. Technology Owner will also be handling the packaging and the labelling according to SOP. Technology Owner will deliver the products to The Partnership upon the order made.	Technology Owner	Month 3 – 12	i. ii.	Technology Owner to deliver product to The Partnership (including list of SOP and Quality Control (QC); Manufacturing SOP by Technology Owner	10%	

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	Based on the feedback from technology and market validation, Technology Owner and The Partnership will conduct discussion on product improvement including packaging and labelling. Technology Owner will guide and advise on technical improvement.		Month 3 - 12	<ul> <li>i. Discussion/meeting request by The Partnership;</li> <li>ii. Continuous quality improvement by Technology Owner</li> </ul>	15%
4.	Quality Control				1.50/
	Technology Owner to carry out quality control procedures on the product at the facility of the appointed manufacturer according to SOP to ensure product samples meets the specifications, safe to use and merchantable quality along the process of validating the technology and market.	Technology Owner	Month 3 - 5	<ul> <li>i. Discussion/meeting request by The Partnership;</li> <li>ii. Technology Owner to provide SOP for Quality Control and reports.</li> </ul>	
5.	Marketing				
	Technology Owner to share any contacts that might be a potential customer and join The Partnership during exhibition (if any, with maximum number of 2 exhibitions) as well as meeting with clients.	Technology Owner	Month 3 – 12	<ul> <li>i. List and contacts of potential customers;</li> <li>ii. Join The Partnership for exhibition (if any, with maximum number of 2 exhibitions)</li> </ul>	20%
6.	The Partnership to carry out technology validation on the Product and provide feedback to the Technology Owner on modification and improvements required on the Product	The Partnership	Month 3 – 12	The Partnership to provide the Technology Owner with a report on the technology validation.	Not applicable

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7.	The Partnership to test the improved or modified Product for compliance with the specification and provide further feedback to the Technology Owner.	The Partnership	Month 3 – 12	The Partnership to provide the Technology Owner with further test reports.	Not applicable
8.	<ul> <li>The Partnership to carry out market validation of improved Products.</li> <li>[The Partnership to specify the steps which will be taken to test the market for the Product. To determine how much details the Partnership wishes to insert as this will be binding on them and also revealing their SOP to the Technology Owner. For instance: <ol> <li>Conducting market survey</li> <li>Providing samples to potential customers such as pharmaceutical companies</li> <li>Obtaining feedback from the market</li> <li>Marketing and promotion of the Product</li> <li>Conducting presentation or seminar as a form of promotion</li> <li>Running of an event or participating in an event to promote the Product]</li> </ol> </li> </ul>	The Partnership	Month 3 - 12	The Partnership to provide the Technology Owner with reports of its market validation activities.	Not applicable
9.	The Partnership to test the modified or improved product based on market feedback and to revert to the Technology Owner if such product requires further modification or improvement for Commercialisation.	The Partnership	3 <sup>rd</sup> Month – 12 <sup>th</sup> Month	Not applicable	Not applicable

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°2.

Technology and Market Validation Agreement

## SCHEDULE IV

## **UNDERTAKING**

ИE



## PRIVATE & CONFIDENTIAL

Our Ref: CRDF/025/17(15-06)

20 September 2017

To:

Orca Greentech PLT Block B, Level 1 UPM-MTDC Technology Centre III Universiti Putra Malaysia 43400 Serdang Selangor Darul Ehsan

Attn: Encik Arif bin Md Zain Chief Executive Officer

OFFER TO PROVIDE FUNDING UNDER THE COMMERCIALISATION OF RESEARCH AND DEVELOPMENT FUND TO ORCA GREENTECH PLT (Registration No: LLP0011051-LGN) ("the Partnership") FOR THE SUM OF RM217,100.00 IN RELATION TO COMMERCIALISATION OF G-GREASE - HIGH PERFORMANCE GREASE USING RECYCLED WASTE OIL ("Project").

We respectfully refer to the above-matter and to your funding application dated 15 June 2017.

Relying upon your representations and Statutory Declaration given in your application as well as during our discussions, we are pleased to inform you that the Approval Committee in its meeting Bil. 3/2017 held on 30 August 2017 has agreed to grant the Partnership, funding under the Commercialisation of Research and Development Fund ("the Funding") to fund the technology validation and market validation activities of your Project but subject to the execution of the fund agreement between the Partnership and MTDC ("Fund Agreement").

The terms and conditions relating to the Funding shall be in accordance with the Fund Agreement and are as follows:

#### 1. Formalisation and Execution of the Fund Agreement

Upon your acceptance of our offer, we shall commence discussions with a view to finalise and execute the Fund Agreement at any event no later than 30 days from the acceptance date of this letter of offer ("**this Letter**").





Date: 30.11.2016

Our ref: UMP078/as Your ref: UMP.05/28.15/1/(NA)

Puan Nor Ilma Mustafa Kamal c/o Research & Innovation (P&I) Level 2, Kompleks Pentadbiran Utama, UNIVERSITI MALAYSIA PAHANG 26600 Pekan, Pahang, Malaysia

COURIER

Dear Sirs:

(

## NEW PATENT APPLICATION IN MALAYSIA – PI 2016400017 filed 30 November 2016

	UNIVERSITI MALAYSIA PAHANG
, ibburger	THOME NAME TO DIN PATALLET A
Inventors:	MOHD NAJIB BIN RAZALI et al

We refer to your UMP original instructions on the above dated 7 October 2016.

We are pleased to report that we have discussed the matter with the main researcher, carried out the novelty searches, drafted the patent specification and submitted it for official filing with the Malaysian Intellectual Property Office (MYIPO).

We provide herewith the particulars of the patent application for your quick reference:

- Title of patent application: "BASE OIL MIXTURE AND A PROCESS OF MANUFACTURE THEREOF"
- UMP project title: Recovery of Base Oil from Waste Sludge and Waste Slop Oil
- 3. MYIPO official patent application no.: PI 2016400017
- 4. MYIPO official application date: 30 November 2016

eastcoast ip management (CA-0239724) 34 LRG IM 10/9, Bukit Istana, 25200 Kuantan – Pahang - Malaysia T: 019 695 8798, ec.patent@gmail.com

# EAST

5. Inventors:

(i) MOHD NAJIB BIN RAZALI (ii) ROSLI BIN MOHD YUNUS

MYIPO will issue a Certificate of Filing (CF) along with preliminary report (to ascertain whether the patent application satisfies the formal requirements) in two to four weeks. We will forward the CF to you upon receipt along with a copy of the patent specification as filed.

We will inform you of any development in the patent application in due course. It has been a pleasure to serve you, and we look forward to working with you again.

Please let us know if you have any questions.

Thank you and best regards,

Ainoon Shabirin Registered Patent Agent (Malaysia) PA/2006/0162

OFFICE COPY

## 

Date: 30 November 2016

Our ref: UMP078/AS Your ref: please advise

INTELLECTUAL PROPERTY CORPORATION OF MALAYSIA (MYIPO) A21-GF, 1st & 2nd, Block A, Kuantan Perdana Commercial Centre, Jalan Tun Ismail 1, 25000 Kuantan, Pahang Darul Makmur

BY HAND

Dear Sirs:

## Re: NEW PATENT APPLICATION IN MALAYSIA Applicant: UNIVERSITI MALAYSIA PAHANG Invention: BASE OIL MIXTURE AND PROCESS OF MANUFACTURE THEREOF

We refer to the above matter and submit herewith the following (in duplicate) for your action:

- 1. Form 1;
- 2. Patent specification;
- 3. Form 17;
- 4. Form 22;
- 5. Form 5B; and
- 6. Official fees.

Please confirm all documents are in order for the new patent application. We look forward to receiving your future reports.

Thank you for your kind assistance, and please confirm receipt.

Please let us know if you have any questions or require any clarification.

Best regards,

Kinoon Binti Shabirin Reg. Patent Agent No. 2006/0162



eastcoast ip management (CA-0239724) 34 LRG IM 10/9, Bukit Istana, 25200 Kuantan – Pahang - Malaysia T: 019 695 8798, ec.patent@gmail.com

AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: <u>A Malaysian Educational Institution</u> * <u>Permanent residence or principal place of business: As above</u>	Patents Form No. 1	For Official Use
REQUEST FOR GRANT OF PATENT [Regulation 7(1)]       Patent Regulation 7(1)]         Request received on: *Fee received on: Malaysia       Pressent received on: *Fee received on: *Generative of the prescribed fee.         Please submit this Form in duplicate together with the prescribed fee.       Applicant's or Agent's file reference UMP078/AS         THE APPLICANT (s) REQUEST (s) THE GRANT OF A PATENT IN RESPECT OF THE FOLLOWING PARTICULARS: BASE OIL MIXTURE AND PROCESS OF MANUFACTURE I. TITLE OF INVENTION: THEREOF         II. APPLICANTON(s) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below): UNIVERSITI MALAYSIA PAHANG Name: PEKAN 26800, PAHANG Address H.S.C / Passport No: Xddress for service in Malaysia: AIMOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798         Nationality:       A Malaysian Educational Institution * Permanent-residence-or principal place of business: As above Additional Information (if any)	PATENTS ACT 1983	APPLICATION No.
[Regulation 7(1)]       Kequest received on:         'Fee received on:       'Fee received on:         'To:       The Registrar of Patents Patent Registration Office Kuala Lumpur       Amount:         'Cheque/Postal Order/Money-Order/Draft/Cash No.       Amount:         'Cheque/Postal Order/Money-Order/Draft/Cash No.         Please submit this Form in duplicate together with the prescribed fee.       Applicant's or Agent's file reference UMP078/AS         THE APPLICANT (\$) REQUEST (\$) THE GRANT OF A PATENT IN RESPECT OF THE FOLLowing PARTICULARS:       BASE OIL MIXTURE AND PROCESS OF MANUFACTURE         I.       TITLE OF INVENTION:       THEREOF         II.       APPLICATION(\$) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below):         Name:       UNIVERSITI MALAYSIA PAHANG         Name:       PEKAN 26600, PAHANG         Address       AINOON BINTI SHABIRIN (PA/2006/0162)         NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA.       T: 019-695 8798         Nationality:       A Malaysian Educational Institution         * Permanent residence or principal place of business: As above       Additional Information (if any)		Filing Date :
To: The Registrar of Patents Patent Registrar of Patents		Request received on:
To: The Registrar of Patents Patent Registration Office Kuala Lumpur Malaysia Please submit this Form in duplicate together with the prescribed fee. THE APPLICANT (s) REQUEST (s) THE GRANT OF A PATENT IN RESPECT OF THE FOLLOWING PARTICULARS: BASE OIL MIXTURE AND PROCESS OF MANUFACTURE I. TITLE OF INVENTION: II. APPLICATION(s) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below): UNIVERSITI MALAYSIA PAHANG Name: H.S.C / Passport No: Address for service in Malaysia: AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: A Malaysian Educational Institution * Permanent-residence-or principal place of business: As above Additional Information (if any)		
Patent Registration Office Kuala Lumpur Malaysia Please submit this Form in duplicate together with the prescribed fee. THE APPLICANT (s) REQUEST (s) THE GRANT OF A PATENT IN RESPECT OF THE FOLLOWING PARTICULARS: BASE OIL MIXTURE AND PROCESS OF MANUFACTURE I. TITLE OF INVENTION: THERBOF II. APPLICATION(s) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below): UNIVERSITI MALAYSIA PAHANG Name: PEKAN 26600, PAHANG Address H.S.C / Passport No: Address for service in Malaysia: AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BINITI SHABIRIN (PA/2006/0162) NO 35, LRG IM10/9 BINITI SHABIRIN (PA/2006/0162) NO 35, LRG IM10/9 BINITI SHABIRIN (PA/2006/0162) NO 36, R		Amount:
Kuala Lumpur Malaysia       Applicant's or Agent's file reference UMP078/AS         Please submit this Form in duplicate together with the prescribed fee.       Applicant's or Agent's file reference UMP078/AS         ThE APPLICANT (s) REQUEST (s) THE GRANT OF A PATENT IN RESPECT OF THE FOLLOWING PARTICULARS: BASE OIL MIXTURE AND PROCESS OF MANUFACTURE I. TITLE OF INVENTION: II. APPLICATION(s) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below): UNIVERSITI MALAYSIA PAHANG Name: PEKAN 26800, PAHANG Address         Name: H.S.C / Passport No: Address for service in Malaysia: AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798         Nationality:       A Malaysian Educational Institution * Permanent residence-or principal place of business: As above Additional Information (if any)	0	* Cheque/Postal Order/Money-Order/Draft/Cash No.
Please submit this Form in duplicate deplicant's or Agent's file reference UMP078/AS THE APPLICANT (s) REQUEST (s) THE GRANT OF A PATENT IN RESPECT OF THE FOLLOWING PARTICULARS: BASE OIL MIXTURE AND PROCESS OF MANUFACTURE I. TITLE OF INVENTION: II. APPLICATION(S) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below): UNIVERSITI MALAYSIA PAHANG Name: UNIVERSITI MALAYSIA PAHANG Address H.S.C / Passport No: Address for service in Malaysia: AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LEG IM109 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: A Malaysian Educational Institution * Permanent residence or principal place of business: As above Additional Information (if any)	Kuala Lumpur	
together with the prescribed fee.       UMP078/AS         THE APPLICANT (s) REQUEST (s) THE GRANT OF A PATENT IN RESPECT OF THE FOLLOWING PARTICULARS:       BASE OIL MIXTURE AND PROCESS OF MANUFACTURE         I. TITLE OF INVENTION:       THEREOF         II. APPLICATION(S) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below):       UNIVERSITI MALAYSIA PAHANG         Name:       UNIVERSITI MALAYSIA PAHANG         Address       PEKAN 26600, PAHANG         Address       NO 34, LEG IM109 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA.         T: 019-695 8798       Nationality:         A Malaysian Educational Institution       * Permanent residence or principal place of business; As above         Additional Information (if any)       J & MY 208		
THE APPLICANT (5) REQUEST (5) THE GRANT OF A PATENT IN RESPECT OF THE FOLLOWING PARTICULARS: BASE OIL MIXTURE AND PROCESS OF MANUFACTURE I. TITLE OF INVENTION: II. APPLICATION(5) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below): UNIVERSITI MALAYSIA PAHANG Name: PEKAN 26800, PAHANG Address H.S.C / Passport No: Address for service in Malaysia: AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: A Malaysian Educational Institution * Permanent residence or principal place of business: As above Additional Information (if any)		
PARTICULARS: BASE OIL MIXTURE AND PROCESS OF MANUFACTURE I. TITLE OF INVENTION: THEREOF II. APPLICATION(S) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below): UNIVERSITI MALAYSIA PAHANG Name: PEKAN 26600, PAHANG Address H.S.C / Passport No: Address for service in Malaysia: AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: A Malaysian Educational Institution * Permanent residence-or principal place of business: As above Additional Information (if any)		
I. TITLE OF INVENTION:       THEREOF         II. APPLICATION(S) (the data concerning each applicant must appear in this box or, if the space is insufficient, in the space below):         UNIVERSITI MALAYSIA PAHANG         Name:		GRANT OF A PATENT IN RESPECT OF THE FOLLOWING
space is insufficient, in the space below): UNIVERSITI MALAYSIA PAHANG Name: PEKAN 26600, PAHANG Address H.S.C / Passport No: Address for service in Malaysia: AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: Adatasian Educational Institution * Permanent residence or principal place of business: As above Additional Information (if any)		
Name:       PEKAN 26600, PAHANG         Address       H.S.C / Passport No:         Address for service in Malaysia:       AINOON BINTI SHABIRIN (PA/2006/0162)         NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA.       T: 019-695 8798         Nationality:       A Malaysian Educational Institution         * Permanent residence or principal place of business: As above       Additional Information (if any)         Additional Information (if any)       Diterimation (if any)		
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H.S.C / Passport No: Address for service in Malaysia: AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: <u>A Malaysian Educational Institution</u> * Permanent residence or principal place of business: As above Additional Information (if any) Ditermina 3 0 KNV 2016 Chargen 2 Ar The Participal Place of Principal Place of P	Addross	PEKAN 26600, PAHANG
Address for service in Malaysia: AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: <u>A Malaysian Educational Institution</u> * Permanent residence or principal place of business: As above Additional Information (if any) Ditermat 3 0 NoV 2016 Compare 2 or Tarter Pagasage in the formation		
AINOON BINTI SHABIRIN (PA/2006/0162) NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: <u>A Malaysian Educational Institution</u> * <u>Permanent residence or principal place of business: As above</u> Additional Information (if any)	H.S.C / Passport No:	
NO 34, LRG IM10/9 BUKIT ISTANA, INDERA MAHKOTA, 25200 KUANTAN, PAHANG, MALAYSIA. T: 019-695 8798 Nationality: <u>A Malaysian Educational Institution</u> * <u>Permanent residence or principal place of business: As above</u> Additional Information (if any)	Address for service in Malaysia:	
* Permanent residence or principal place of business: As above Additional Information (if any) Diferimed 3.0 NOV 2016 Caesangen 2 on Timu: Persidence Hate Interview	NO 34, LRG IM10/9 E 25200 KUAN	BUKIT ISTANÀ, INDERA MAĤKOTA, ITAN, PAHANG, MALAYSIA.
Additional Information (if any) Dilerima 5 0 NNV 2016 Cawangan 201 Time Perbadapan Farta ana Unartia ana Unartia	Nationality: <u>A Malaysian Edu</u>	cational Institution
Dilerima 5 0 NOV 2016 Cawangan 200 Yind: Perbadapan Harta Interac Maarin Perbadapan Harta Interac Maarin Perbadapan Harta Interac Maarin	* Permanent residence or principa	al place of business: As above
Cawangan 2on Yimu: Perbadupan Hada Perbadupan Kada Induras Masayaha	Additional Information (if any)	
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	INVENTOR:				
	Applicant is the inventor	r	,	Yes	No 🗸
	If the applicant is not the	e inventor :			L1
Name		ID NAJIB BIN RAZ LI BIN M <mark>OHD YU</mark> N			
	Address of inventors:	UNIVERSITI MALA GAMBANG, MALA			
	A statement justifying th			panies this	Form:
				Yes 🗸	No
Additio	nal Information (if any)	· · · · · · · · · · · · · · · · · · ·			
IV.	AGENT OR REPRESENTAT	TIVE:			
	Applicant has appointed	a patent agent in acc	ompanying Form	No. 17	Yes 🗸
					No
	Agent's Registration No		······································		
	Applicants have appoint		Snapirin		2010 V Datasta - 22.000 V Datasta
	to be their common repr	resentative.			·
V.	DIVISIONAL APPLICATION:				
	This application is a divi	1 1 1 1 1			
	The benefit of the filing of		priority date		
	of the initial application		nuch as the sul	oject-matter	of the prese
		in the initial application	on identified below	N:	
	application is contained				
	application is contained Initial Application No.:				

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	SCLOSURES TO BE DISREGARDED FOR PRIOR ART PURPOSES:	
Ad	Iditional information is contained in supplemental box:	[]
(a)	Disclosure was due to acts of applicant or his predectitle	essor in
	Date of disclosure:	<b></b> _
(b)	Disclosure was due to abuse of rights of applicant or his predecessor in title	
As	Date of disclosure: statement specifying in more detail the facts concerning the	Yes
dis	closure accompanies this Form:	
		No
Additional	Information (if any)	
VII. F	PRIORITY CLAIM (if any): NIL	
Т	The priority of an earlier application is claimed as follows:	
C	Country (if the earlier application is a regional or internationa office with which it is filed):	I application, indicate the
	iling Date:	
	Application No.:	
	Symbol of the International Patent Classification :	
lf	f not yet allocated, please tick	
Т	The priority of more than one earlier application is claimed:	
	Yes	No
т	he certified copy of the earlier application (s) accompanies th	is Form:
	Yes	No
lf	No, it will be furnished by	
dditional	Information (if any)	Diterima 3 0 Mily 2016
	<u>,</u>	10-2 Convergiant Zon Timu: Convergiant Zon Harta
		VSA Inteles Manaysia 📈

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VIII. CHECK LIST: A. This application contains the following: 1. request 4 2. description 14 sheets 3. claim 1 sheets 4. abstract 1 sheets 5. drawings sheets 1 Total 21 sheets B. This Form, as filed, is accompanied by the items checked below: (a)signed Form No. 17  $\sqrt{}$ (b) declaration that inventor does not wish to be named in the patent statement justifying applicant's right to the patent *(c)*  $\sqrt{}$ (d)statement that certain disclosures be disregarded priority document (certified copy of earlier application) (e) (f)cash, cheque, money order, banker's draft or postal order for  $\sqrt{}$ the payment of application fee other documents (specify) (g) IX. SIGNATURE: Kindon 30.11.2016 Ainoon Binti Shabirin \*\* (Applicant/Agent) (Date) If Agent, indicate Agent's Registration No.: PA/2006/0162 For Official Use 1. Date application received : 2. Date of receipt of correction, later filed papers or drawings completing the application:



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\* Delete whichever does not apply.

P.	atents Form No. 5B ATENTS ACT 1983	APPLICATION NO:	For Official Use		
0	EQUEST FOR DEFERMENT OF FILING F REQUEST FOR EXAMINATION OR ROVISION OF INFORMATION	Filing Date:			
(F	Regulations 27b(1) and 45(3))	Trequest received of	1		
T	o: The Registrar of Patents Patents Registration Office Kuala Lumpur, Malaysia	Date of mailing			
Ple	ase submit this Form in duplicate	Applicant's or Agent	's file reference : UMP078/AS		
1.	IN THE MATTER OF:				
	Patent Application No.: Certificate Application No.:	Filing Date: Filing Date:			
11.	APPLICANT(S) UNIVERSITI N	IALAYSIA PAHANG			
	Name : 26600 PEKAN Address :	, PAHANG			
111.	REQUEST:				
	*(a) The applicant requests defem Section 29a (1) or 29A (2) of th		quest for examination referred to		
	Particulars of the *patent/certif in Section 29A(2) of the Patent		ntially the same invention referred		
	Name of Country: Application No.: Date Application Ledged:				
	I-certify that a *patent/certificat in respect of the above applica		in or is not availat		
	Of (b) The employeet requests deform	ant of the provision of the	*information/currenting degument		
]	(b) The applicant requests defermed to in Section 29A (4) o		*information/supporting document		
and a first state of the state	I certify that a *information/su application.	pporting document is no	ot available in respect of the abo		
VI.	ADDITIONAL INFORMATION accomp	anies this Form: Yes	No 🗵		
	SIGNATURE	•	30.11.2016		
	AINOON BINTI SHAE ** (Applicant-/ Agent)		(Date)		
( V.	If Agent, indicate Agent's Registration h		3 U FOV 2016		
	Delete whichever does not apply Type name under signature and delete whiche	ver does not apply	Ciswangan Zon Timu Perhadarian Harta Unterik Malaysia		
			×210183		

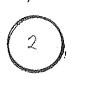
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UMP077 PT Cover Letter Confirmation		For Official Use
Patents Form No. 17 PATENTS ACT 1983 APPOINTMENT OR PATENT AGENT (Regulations 45B (2)	CHANGE OF	APPLICATION NO. : Filing date : Received on :
To : The Registrar of Patents Registr Kuala Lumpur, I	ation Office	
Date of Mailing		
Please submit one co	ppy of this Form	Applicant's or Agent's file reference: UMP078/AS
*I/ We the under:	signed ,	
Name : UNIVE	ERSITI MALAYSIA PA	HANG
Address : 26600	PEKAN, PAHANG	
appoint Name :	NO 34, LRG IM1	I BINTI SHABIRIN (PA2006/0162), 10/9 BUKIT ISTANA, INDERA MAHKOTA, WANTAN, PAHANG, MALAYSIA.
to act as *my/our	agent in connection w	T: 019-695 8798 vith Malaysian Patent Application entitled
BA	SE OIL MIXTURE A	ND PROCESS OF MANUFACTURE THEREOF
	otices, requisitions an	*my/our behalf in connection with that/those matter(s), and d communications relating thereto be sent to the agent at the
Any previous app	pointment in respect of	the same matter(s) is revoked.
		providence .
**SIGNATURE	NOR / PENO	HMA BINTI MUBTAFA KAAAAL XONG PENDAETAR (HARTA BITELEK)
DATE:		BAT TRABALAN NAIB CANSELOR KELIDIKABLE INOVASI) ERSITTMALAYSIA PAHANG
NAME:	Lebu 26300 Pahan	HRAYA TUN BAZAK GAMBANG, KUANTAN NG DARUL MAKMUR
DESIGNATION:	TEL: 0	19-549 3338 FAKS: 08-548 3324
·····		ete whichever does not apply der signature and delete whichever does not apply
		Partiality mains

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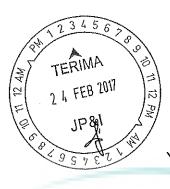
Patents Form No. 22 PATENTS ACT 1983	For Official Use
STATEMENT JUSTIFYING THE APPLICANT'S RIGHT TO A PATENT/CERTIFICATE (Regulations 10(2)) To : The Registrar of Patents	APPLICATION NO.: 2016400017 Filing Date : Request received on :
Patents Registration Office Kuala Lumpur, Malaysia	
	Date of mailing
Please submit this Form in duplicate	Applicant's or Agent's file reference UMP078/AS
I. IN THE MATTER OF : Patent Application No. : Certificate Application No. :	Filing Date : Filing Date :
II. TITLE OF INVENTION: BASE OIL MIXTU THEREOF	URE AND PROCESS OF MANUFACTURE
III. APPLICANT (S) : Name : <u>UNIVERSITI MALAYSIA PAHAN</u> Address : <u>26600 PEKAN, PAHANG</u>	<u>\G</u>
IV. I/we believe that the inventor(s)/innovator(s	s) of the above mentioned application is as follows :
Name of Inventor(s):	: MOHD NAJIB BIN RAZALI ROSLI BIN MOHD YUNUS
Address of Inventor(s): UNIVE	ERSITI MALAYSIA PAHANG, GAMBANG, MALAYSIA
V. Statement justifying the applicant's right to	a patent/certificate :
The Applicant entitles the right to grant invention by virtue of employment.	a patent in relation to the above captioned
VI. ADDITIONAL INFORMATION accompanies	this Form :
Yes	No x
VII. SIGNATURE	30.11.2016
** (Applicant/Agent) AINOON BINTI SHABI	
If Agent, indicate Agent's Registration No. PA	V/2006/0162
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AST



Date: 22 Feb 2017

Our ref: UMP080/as Your ref: UMP.05/28.15/1/(NA)

Puan Nor Ilma Mustafa Kamal c/o Research & Innovation (P&I) Level 2, Kompleks Pentadbiran Utama, UNIVERSITI MALAYSIA PAHANG 26600 Pekan, Pahang, Malaysia

COURIER

Dear Sirs:

NEW PATEN	IT APPLICATION IN MALAYSIA - PI 2017	7400003 FILED 22 FEB 2017
Applican <mark>t:</mark>	UNIVERSITI MALAYSIA PAHANG	
Inventors:	MOHD NAJIB RAZALI et al	

We refer to your UMP instructions on the above dated 16 December 2017.

We are pleased to report that we have discussed the matter with the main researcher, carried out the novelty searches, drafted the patent specification and submitted it for official filing with the Malaysian Intellectual Property Office (MYIPO).

We provide herewith the particulars of the patent application for your quick reference:

- 1. Title of patent application: "A Thickening Agent for Grease Formulation and Associated Grease Formulation Using the Same"
- 2. UMP project title: Formulation of Multipurpose Grease from Industrial Waste
- 3. MYIPO official patent application no.: PI 2017400003
- 4. MYIPO official application date: 22 February 2017

eastcoast ip management (CA-0239724) 34 LRG IM 10/9, Bukit Istana, 25200 Kuantan, Pahang - Malaysia T: 019 695 8798, ec.patent@gmail.com



5. Inventors:

## MOHD NAJIB BIN RAZALI MOHD AIZUDIN BIN ABD AZIZ ROSLI BIN MOHD YUNUS MUSFAFIKRI BIN MUSA

MYIPO will issue a Certificate of Filing (CF) along with preliminary report (to ascertain whether the patent application satisfies the formal requirements) in two to four weeks. We will forward the CF to you upon receipt along with a copy of the patent specification as filed.

We will inform you of any development in the patent application in due course. It has been a pleasure to serve you, and we look forward to working with you again.

Please let us know if you have any questions.

Thank you and best regards,

In com

Ainoon Shabirin Registered Patent Agent (Malaysia) PA/2006/0162

Date: 22 FEB 2017

Our ref: UMP080/AS Your ref: please advise

EAST

OFFICE COPY

INTELLECTUAL PROPERTY CORPORATION OF MALAYSIA (MYIPO) A21-GF, 1st & 2nd, Block A, Kuantan Perdana Commercial Centre, Jalan Tun Ismail 1, 25000 Kuantan, Pahang Darul Makmur

BY HAND

Dear Sirs:

٤.

Re: NEW PATENT APPLICATION IN MALAYSIA Applicant: UNIVERSITI MALAYSIA PAHANG Invention: A THICKENING AGENT FOR GREASE FORMULATION AND ASSOCIATED GREASE FORMULATION USING THE SAME

We refer to the above matter and submit herewith the following (in duplicate) for your action:

- 1. Form 1;
- 2. Patent specification;
- 3. Form 17;
- 4. Form 22;
- 5. Form 5B; and
- 6. Official fees.

Please confirm all documents are in order for the new patent application. We look forward to receiving your future reports.

Thank you for your kind assistance, and please confirm receipt.

Please let us know if you have any questions or require any clarification.

Best regards,

Curr

Ainoon Binti Shabirin Reg. Patent Agent No. 2006/0162



eastcoast ip management (CA-0239724) 34 LRG IM 10/9, Bukit Istana, 25200 Kuantan – Pahang - Malaysia T: 019 695 8798, ec.patent@gmail.com

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To : The Registrar of Patent Patents Registration O Kuala Lumpur, Malays	ffice			
Date of Mailing				
Please submit one copy of t	his Form	Applicant's or Agent's file	reference: UMP080//	AS
*I/ We the undersigned	,			
Name : UNIVERSITI	MALAYSIA PA	HANG	$  g_{i}   =   g_{i}  $	
Address : 26600 PEKA	n, pahang	<u> </u>	•	
appoint Name : N	IO 34, LRG IM1	BINTI SHABIRIN (PA200 10/9 BUKIT ISTANA, INDE CUANTAN, PAHANG, MAL T: 019-695 8798	RA MAHKOTA,	
A THICKENING AGENT	FOR GREASE	vith Malaysian Patent App FORMULATION AND		SE
FORMULATION USING	THE SAME			
request that all notices abovementioned said a	, requisitions an ddress.	*my/our behalf in conne nd communications relatin	g thereto be sent to the	natter(s), and e agent at the
Any previous appointme	ent in respect of	f the same matter(s) is rev	oked.	
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**SIGNATURE	NOR PEN PEN	HAA-BINTRANUGTAFA-HAMAL OLONG PENDAFTAR (HARTA DITE BAT TIMBALAN NAIB CANSELOR	LEK)	
DATE:	(PEN UNIV	RELIDIKAN KINOVABI ERSITT MALAYSIA PAHANG		
NAME:	2530	MRAYA TUN RAZAK O GAMBANG, KUAATAN ING DARUL MAKIMUR		
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				P. N. Martin

Contraction

Patents Form No. 22 PATENTS ACT 1983	For Official Use
STATEMENT JUSTIFYING THE APPLICANT'S	
RIGHT TO A PATENT/CERTIFICATE (Regulations 10(2))	Filing Date :
	Request received on :
To: The Registrar of Patents Patents Registration Office	the second se
Kuala Lumpur, Malaysia	·
	Date of mailing
Please submit this Form in duplicate	Applicant's or Agent's file reference
	UMP080/AS
I. IN THE MATTER OF :	
Patent Application No. :	Filing Date :
Certificate Application No. :	Filing Date :
II. TITLE OF INVENTION: A THICKENIN ASSOCIATED GREASE FORMULATIO	NG AGENT FOR GREASE FORMULATION AND
ASSOCIATED GREASE FORWIOLATIN	ON USING THE SAME
III. APPLICANT (S) :	
Name : UNIVERSITI MALAYSIA PA	
Address : 26600 PEKAN, PAHANG	
I/we believe that the inventor(s)/innova	tor(s) of the above mentioned application is as follows :
	tor (5) of the above mentioned application is as follows .
	MOHD NAJIB BIN RAZALI
	MOHD AIZUDIN BIN ABD AZIZ
	ROSLI BIN MOHD YUNUS MUSFAFI≭RI BIN MUSA
	MUSPAPIERI BIN MUSA
Address of Inventor(s):	NIVERSITI MALAYSIA PAHANG, PEKAN, MALAYSIA
V. Statement justifying the applicant's rig	bt to a patent/certificate
	rant a patent in relation to the above captioned
invention by virtue of employment.	Tant a patent in relation to the above capitolled
VI. ADDITIONAL INFORMATION accompa	anies this Form:
Yes	Nox
J' Mar	22.2.2017
VII. SIGNATURE	
** (Applicant/Agent) AINOON BINTI SH	
If Agent, indicate Agent's Registration No	o. <u>PA/2006/0162</u>
	the second se

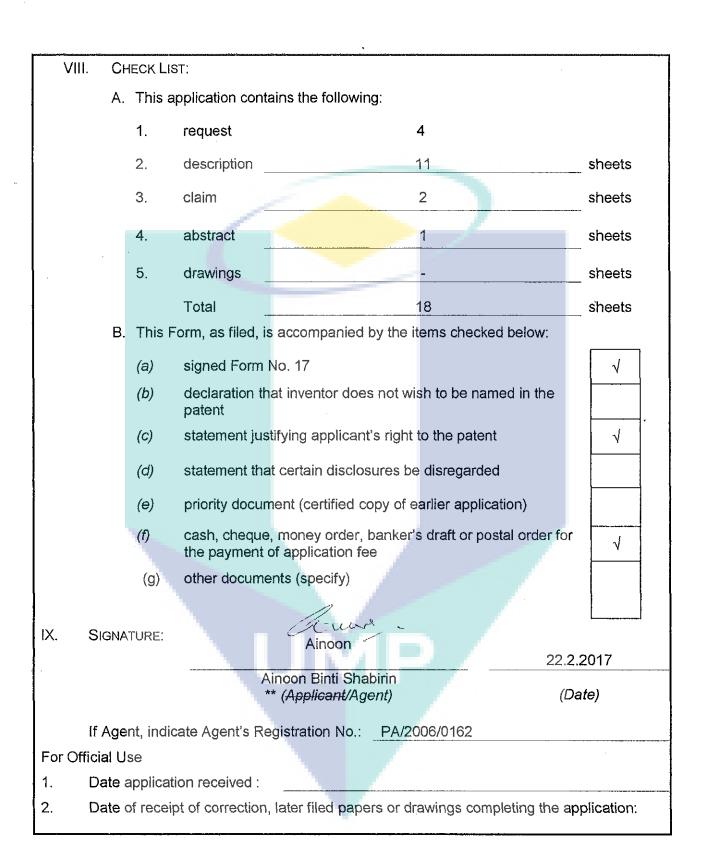
Patents Form No. 1	For Official Use
PATENTS ACT 1983	APPLICATION No.
	Filing Date :
REQUEST FOR GRANT OF PATENT	Request received on:
[Regulation 7(1)]	*Fee received on:
	Amount:
To: The Registrar of Patents Patent Registration Office	* Cheque/Postal Order/Money Order/Draft/Cash No.
Kuala Lumpur	
Malaysia	
Please submit this Form in duplicate together with the prescribed fee.	Applicant's or Agent's file reference UMP080/AS
PARTICULARS:	GRANT OF A PATENT IN RESPECT OF THE FOLLOWING EXENING AGENT FOR GREASE FORMULATION SSOCIATED GREASE FORMULATION USING THE
II. APPLICATION(S) (the data conce space is insufficient, in the space	erning each applicant must appear in this box or, if the e below): UNIVERSITI MALAYSIA PAHANG
Address	PEKAN 26600, PAHANG
· · · · · · · · · · · · · · · · ·	
H.S.C / Passport No:	
Address for service in Malaysia:	
NO 34, LRG IM10/9 25200 KUAI	ITI SHABIRIN (PA/2006/0162) BUKIT ISTANA, INDERA MAHKOTA, NTAN, PAHANG, MALAYSIA. T: 019-695 8798
Nationality: A Malaysian Ed	ucational Institution
* Permanent residence or princip	bal place of business: As above
Additional Information (if any)	13. 15 15 Ja
	a Julia Lina IV

	INVENTOR:					[]	·
	Applicant is the	inventor			· Yes		No 🗸
	If the applicant i	s not the	e inventor :				· .
	Name of invento	ors :	_				:
		7	MOHD NA. MOHD AIZUI	JIB BIN RAZ			
		· /		MOHD YUN			
				KRI BIN MU			
	Address of inve	entors:		I MALAYSIA F HANG - MALA			
	A statement just	ifying th	e applicant's ri	ight to the pat	ent accompa	nies this F	orm:
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dditio	onal Information (	f any)					
IV.	AGENT OR REPR	ESENTAT	IVE:				
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	to be their comm	non repr	esentative.	VT-	. /		
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V.	DIVISIONAL APPL	ICATION:					
	This application	is a divi	sional applicat	ion			
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VI. DISCL	LOSURES TO BE DISREGARDED FOR PRIOR ART PURPOSES:	
Additi	tional information is contained in supplemental box:	
(a)	Disclosure was due to acts of applicant or his predecessor in title	
	Date of disclosure:	
(b)	Disclosure was due to abuse of rights of applicant or his predecessor in title	
	Date of disclosure:	
A stat	atement specifying in more detail the facts concerning the . Yes	
discla	osure accompanies this Form:	
	No	
Additional Inf	nformation (if any)	
VII. Pri	IORITY CLAIM (if any): NIL	
The	e priority of an earlier application is claimed as follows:	
Cou	ountry (if the earlier application is a regional or international application, indicate thice with which it is filed):	ıe
Filir	ing Date:	
Арр	plication No.:	
Syn	mbol of the International Patent Classification :	
lf no	not yet allocated, please tick	
The	e priority of more than one earlier application is claimed:	
	Yes No	
The	e certified copy of the earlier application (s) accompanies this Form:	
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	Yes No	
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## A THICKENING AGENT FOR GREASE FORMULATION AND ASSOCIATED GREASE FORMULATION USING THE SAME

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### 5 FIELD OF INVENTION

The present invention relates to a novel thickening agent for grease formulation and subsequently a grease formulation comprising said thickener.

#### BACKGROUND OF INVENTION

Grease is used in various engineering applications. A grease exhibits shear-thinning rheological properties, which means that the viscosity of the grease reduces under shear. A grease typically comprises the base oil such as mineral oil, and a thickening agent or a thickener. Under non-shear conditions, the thickener acts as a sponge-like matrix that holds the base oil. As a result, the grease exhibits the characteristics of a solid or semisolid. The base oil may be released from the matrix both under static or dynamic conditions. In particular, when a shear force is applied, the base oil separates from the thickener matrix and results in the grease exhibiting lubricating properties.

The base oil is important in determining the properties of the grease. The interaction of the base oil and the thickener is also important in determining, for example, the longevity of a grease when used as a lubricant. Greases for bearings have been

described in EP 0700986, EP 0795597 and EP 0795598. In these documents, a polymer is used as the thickener in the grease composition. The base oil chosen is the conventional mineral oil, a synthetic hydrocarbon oil or an ester oil.

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As described in EP 0700986, a typical method for the manufacture of a grease is to form a homogeneous mixture of the desired base oil and thickener, and then to rapidly cool or quench it. This may be achieved by pouring the composition onto a cooling table.

Thickeners based on polymers include polycarbamides (polyureas) and

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polytetrafluoroethylene. Thickeners based on urea compounds are disclosed in WO
2011/020863 A1. Greases comprising polymeric thickeners are disclosed in WO
2012/076025 A1. Inorganic thickeners such as bentonite, amorphous hydrophilic silicon
oxide particles and silica gel are also commonly used. Preferably silica particles having
a mean particle size in the range of 5 to 50 nm can be used as described in US
2012/149613 A1.

French Application FR 2 172 080 describes particularly stable greases thickened with aluminum soaps, as does application FR 2 012 238, which also stresses their water resistance. EP 0661378 discloses examples of greases thickened with complex aluminum soaps containing molybdenum dithiocarbamate (MoDTC), alone or in combination with zinc dithiophosphate (ZnDTP). These compositions contain no graphite. Their EP performances are mediocre, particularly those of the grease containing only MoDTC.

The present invention aims to address at least some of the problems associated with the prior art or at least to provide an alternative to the existing products.

20 In particular, there is a need for an alternative thickening agent or thickener which is cost-effective and which use is advantageous from an environmental perspective.



Additionally, there is a need for a grease composition based on the alternative thickener. Depending on the composition, the grease could be formulated for machinery or automotive applications. Due to the environmentally safe contents, the prepared grease can minimize environmental issues as well as reduce manufacturing costs.

5 Other objects and features of the present invention will become apparent from the following detailed description, considered in conjunction with the accompanying drawing figures. It is to be understood, however, that the drawings are designed solely for the purpose of illustration and not as a definition of the limits of the invention, for which reference should be made to the appended claims.

### 10 SUMMARY OF INVENTION

In accordance with the present invention, there is provided a thickening agent for grease – formulation, comprising red gypsum.

Preferably, the thickening agent comprises a mixture of red gypsum, NaOH, and Al2(OH)3.

Most preferably, the thickening agent comprises a mixture of red gypsum, NaOH, Al2(OH)3, and fumed silica.

Still according to the present invention, there is provided a grease formulation comprising;

(a) a base oil;

20 (b) a thickener; and

(c) additive/s,



wherein the base oil is used oil and/or waste emulsion, and wherein the thickener is in an amount of 10 to 30% by weight based on the total amount of the base oil, and wherein the thickener comprises red gypsum.

In one embodiment, the base oil is pre-treated used oil and/or a waste emulsion.

5 Preferably, the used oil includes refined used automotive and/or machinery lubricant.

Preferably, the waste emulsion includes automotive and/or industrial coolant. Preferably, the thickener is a mixture of NaOH, Al2(OH)3 and red gypsum. Most preferably, the thickener is a mixture of NaOH, Al2(OH)3, red gypsum and fumed silica.

10 Preferably, the additive is in an amount of 5 to 10% by weight based on the total amount of the base oil.

Preferably, the additive is iron octoate.

Preferably, the additive is molybdenum disulphide.

- Most preferably, the additive is a mixture of iron octoate and molybdenum disulphide.
- 15 The particular features and advantages of the invention as well as other objects will become apparent from the following description.

#### DETAILED DESCRIPTION OF INVENTION

20

Grease is a semi-solid composition comprising three (3) major components namely base oil, a thickening agent or thickener, and an additive. The present invention is directed to the grease formulation using base oil from recovered oil from industrial waste emulsion, used oil from automotive industry, and red gypsum as thickener. At present, these waste oil and emulsion have posed industrial problems because they require complex disposal treatment and processing.

The present work, for the first time in industry describes the use of red gypsum as a thickening agent or thickener. Red gypsum is completely different in chemical and physical properties compared to the commercially used thickener, namely fumed silica. Currently, red gypsum is classified as an industrial by-product or waste, and the disposal of red gypsum using land filling method and storage of gypsum on land has posed negative impacts to the environment. Red gypsum, is a waste material from the production of titanium dioxide which contains between 60-90 % CaSO4.2H2O 3-35 % iron II and iron III oxide/hydroxides, expressed as FeOH3 0.3 to 2 % TiO2 0.1 to 4% SiO2 0.1 to 0.8 % Na2O 0.1 to 5 % CaCO3 0.1 to 0.4 % MgO 0.1 to 0.2 % potassium, calculated as K2O and 0.1 to 0.2 % phosphorous, calculated as P2O5. The current and commercial use of red

Additionally, molybdenum disulphide is used as an additive and iron octoate as antioxidant agent.

Hence in accordance with the present invention, there is provided a thickener or thickening agent for grease formulation, comprising red gypsum.

Preferably, the thickening agent comprised a mixture of red gypsum and NaOH, Al2(OH)3.

Most preferably, the thickening agent comprises a mixture of red gypsum, NaOH,
 Al2(OH)3, and fumed silica.

Additionally, the present invention describes a grease formulation comprising,

(a) a base oil;

gypsum is in soil amendments.

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(b) a thickener; and

(c) additive/s,

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wherein the base oil is used oil and/or waste emulsion, and wherein the thickener is in an amount of 10 to 30% by weight based on the total amount of the base oil, and wherein the thickener comprises red gypsum.

Preferably, the grease formulation base oil is pre-treated used oil and/or a waste emulsion.

Preferably, the used oil includes refined used automotive and/or machinery lubricant. Preferably, the waste emulsion includes automotive and/or industrial coolant.

- 10 Preferably, the thickener is a mixture of NaOH, Al2(OH)3 and red gypsum.
  - Most preferably, the thickener is a mixture of NaOH, Al2(OH)3, red gypsum and fumed silica.

Most preferably, the additive is in an amount of 5 to 10% by weight based on the total amount of the base oil.

15 Preferably, the additive is iron octoate.

Preferably, the additive is molybdenum disulphide.

Most preferably, the additive is a mixture of iron octoate and molybdenum disulphide.

The preparation method of the grease formulation of the present invention will be described via the following experimental methods.

### 20 EXPERIMENTALS

### **Materials and Methods**

### Chemicals

Base oil was recovered oil from waste emulsion, used oils (waste lubricant oil) taken from manufacturing and automotive industry. 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 (3) components that needed to be mixed to produce iron octoate paste as an anti-oxidant agent are octoic acid (purchased from Acros with 99% purity), caustic soda with 96% purity was purchased from Merck and ferrous sulphate that also purchased from Merck, has 99.5% purity.

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10 For soap based thickener, the raw materials used were sodium hydroxide, aluminium hydroxide and cooking oil. Sodium hydroxide and aluminium hydroxide were in solid form purchased from Sigma Aldrich whereas the cooking oil used was vegetable oil purchased from convenient store. For the non-soap based thickener, fumed silica was obtained from FKKSA laboratory.

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### Formulation of Iron Octoate as Anti-Oxidant Agent

There are generally three (3) steps in iron octoate preparation. The first step is saponification. Caustic soda is dissolved in 50ml deionized water. Then, octoic acid is added with stirring and heating by hot-plate magnetic stirrer for two (2) hours to produce soluble sodium soap. Temperature is maintained at 80-90oC. The second step is precipitation. 200ml of 15% solution of ferrous sulphate is added to iron octoate precipitate. The reaction time is 10 hours as the temperature is maintained. The third step is drying process. Slurry is filtered and washed with vacuum filter, then dried in an oven at 65°C for 90 minutes. The dried precipitations is then crushed to powder.

### **Formulation of grease**

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There are generally four (4) steps 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 desired amount of Sodium hydroxide, aluminium hydroxide, fumed silica and red gypsum is slurred with ½ of the require amount of waste used oils with a continuous stirring. Then the third step is continuous mixing and heating at a constant temperature. Finally the forth step is addition and continuous mixing. The rest namely ½ of the waste oils, iron octate and molybdenum disulphide add slowly into the smooth paste. The mixing and heating process is continued for 6 hours. However, it should be obvious to one skilled in the industry that these steps could be modified or altered to achieve the desired product

The produced grease formulation is then be analysed using American Society for Testing and Materials (ASTM) to determine its penetration test, dropping point test, and amount of ail generated. These tests are important as comparison would be made to comparisolu-

15 of oil separated. These tests are important so comparison could be made to commercially available grease formulation.

### **Sample Analysis**

### ASTM (American Society of Testing and Materials) testing

20 ASTM testing's done to determine the stability, quality and properties for general usage. The tests 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)

depending on the targeted properties.

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 0 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 hasbeen 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 millimetre. The further the cone penetrates the grease, the higher the penetration result and the softer the grease.

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### 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 happens 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)

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Oil separation test determines 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.



This produced grease formulation 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. NLGI consistency and stability grades are in range between 0 to 6.

5 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.

### 10 Results

Specification of Grease

TEST	метнор	RESULT
Cone Penetration	ASTM D-217	340
Color	ASTM D-1500	Dark grey
Appearance	ASTM D-4176	Rough
Dropping Point	ASTM D-2265	No Melting after 240 deg C
Worked 60x Stroke	ASTM D-217	295
NLGI Grade		1

As observed the above, the produced grease formulation is on par with the commercial grease formulations.



Thus, while there have been shown and described and pointed out novel features of embodiments of the present invention, it will be understood that various omissions and substitutions and changes in the form and details of the disclosed invention may be made by those skilled in the art without departing from the scope of the invention.

5 It is also to be understood that the following claims are intended to cover all of the generic and specific features of the invention herein described and all statements of the scope of the invention which, as a matter of language, might be said to fall there between.



1. A thickening agent for grease formulation, comprising red gypsum.

2. A thickening agent of claim 1, comprising a mixture of red gypsum and NaOH,

5 Al2(OH)3.

3. A thickening agent of claim 1, comprising a mixture of red gypsum, NaOH, Al2(OH)3, and fumed silica.

4. A grease formulation comprising;

(a) a base oil;

10 (b) a thickener; and

(c) additive/s,

wherein the base oil is used oil and/or waste emulsion, and wherein the thickener is in an amount of 10 to 30% by weight based on the total amount of the base oil, and wherein the thickener comprises red gypsum.

15 5. A grease formulation of claim 4, wherein the base oil is pre-treated used oil and/or a waste emulsion.

6. A grease formulation of claim 4, wherein the used oil includes refined used automotive and/or machinery lubricant.

7. A grease formulation of claim 4, wherein the waste emulsion includes automotive

20 and/or industrial coolant.



A grease formulation of claim 1 wherein the thickener is a mixture of NaOH,
 Al2(OH)3 and red gypsum.

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A grease formulation of claim 1, wherein the thickener is a mixture of NaOH,
 Al2(OH)3, red gypsum and fumed silica.

5 10. A grease formulation of claim 1, wherein the additive is in an amount of 5 to 10% by weight based on the total amount of the base oil.

11. A grease formulation of claim 6, wherein the additive iron octoate.

12. A grease formulation of claim 1, wherein the additive is molybdenum disulphide.

13. A grease formulation of claim 1, wherein the additive is iron octoate and molybdenum disulphide.



### ABSTRACT

# A THICKENING AGENT FOR GREASE FORMULATION AND ASSOCIATED GREASE FORMULATION USING THE SAME

The present invention describes an alternative thickening agent or thickener for grease formulation which is cost-effective and which use is advantageous from an environmental perspective. Additionally, the present invention describes a grease formulation using the novel thickener. Depending on the composition, the grease could be formulated for machinery or automotive applications. Due to the environmentally
 safe contents, the prepared grease can minimize environmental issues as well as reduce manufacturing costs.



# SAFETY DATA SHEET

# SECTION 1: PRODUCT AND COMPANY IDENTIFICATION

PRODUCT NAME PRODUCT CODES		: G-Grease : LCG 015				
MANUFACTU ENGINEERIN		: FACULTY OF CHEMICAL AN	ND NATURAL RESOURC	CE		
ADDRESS		: UNIVERSITI MALAYSIA PAH LEBUHRAYA TUN RAZAK, 26300, GAMBANG, PAHANG	,			
EMERGENCY OTHER CALL FAX PHONE		: +6095492928/3326 (OFFICE : : +6095492889	HOUR)			
PRODUCT US	SE	: Lubricating Grease				

# SECTION 2: HAZARDS IDENTIFICATION

### POTENTIAL ACUTE HEALTH EFFECTS :

Slightly hazardous in case of eye contact (irritant), of ingestion. Non-irritant for skin. Non-hazardous in case of inhalation.

**POTENTIAL CHRONIC HEALTH EFFECTS: CARCINOGENIC EFFECTS:** 3 (Not classifiable for human.) by IARC. **MUTAGENIC EFFECTS:** Not available. **TERATOGENIC EFFECTS:** Not available. **DEVELOPMENTAL TOXICITY:** Not available. Repeated or prolonged exposure is not known to aggravate medical condition.

# **SECTION 3: COMPOSITION/INFORMATION ON INGREDIENTS**

PRODUCT DEFINITION	: Mixture
DESCRIPTION	: Mineral oil synthetic hydrocarbon oil complex soap
PHYSICAL STATE	: Semi-Solid
COLOR	: Grey
ODOR	: Neutral

### ENVIRONMENTAL HAZARD :

Very toxic to aquatic organisms, may cause long-term adverse effect in the aquatic environment

### **TOXICOLOGY DATA ON INGREDIENTS :**

#### Not applicable

There are no additional ingredients present which, within the current knowledge of the supplier and in the concentrations applicable, are classified as hazardous to health or the environment.



# **SECTION 4: FIRST AID MEASURES**

**EYES** : Check for and remove any contact lenses. In case of contact, Immediately flush eyes with plenty of water for at least 15 minutes. Occasionally lifting the upper and lower eyelids. Get medical attention if irritation occurs.

SKIN : Wash with soap and water. Get medical attention if irritation develop

**INGESTION:** Wash out mouth with water. Move exposed person to fresh air. Keep person warm and at rest. If material has been swallowed and the exposed person is conscious, give small quantities of water to drink. Do not induce vomiting unless directed to do so by medical personnel. Get medical attention if symptoms occur

**INHALATION:** If inhaled, remove to fresh air. Keep person warm and at rest. if breathing is irregular or if respiratory arrest occurs, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.

**NOTES TO PHYSICIANS OR FIRST AID PROVIDERS:** No action shall be taken involving any personal risk or without suitable training.

**NOTES TO DOCTOR :** No specific treatment. Treat symptomatically. Contact poison treatment specialist immediately if large quantities have been ingested or inhaled

# **SECTION 5: FIRE-FIGHTING MEASURES**

FLAMMABILITY OF THE PRODUCT: : No special fire risk. In case of fire use foam, carbon

dioxide, dry chemical powder and water mist.

- AUTO-IGNITION TEMPERATURE FLAMMABLE LIMITS PRODUCTS OF COMBUSTION
- : Not available. : Not available. : Not available.

EXPLOSION HAZARDS IN PRESENCE OF VARIOUS SUBSTANCES:

Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available.

FIRE FIGHTING MEDIA AND INSTRUCTIONS: SUITABLE EXTINGUISHING MEDIA : Water, Carbon dioxide (CO2) UNSUITABLE EXTINGUISHING MEDIA: None known.

### FIRE-FIGHTERS GUIDELINE:

Don't breathe combustion fumes: fire can form harmful compounds. Do not inhale explosion and combustion gases.

Burning produces heavy smoke.

Promptly isolate the scene by removing all persons from the vicinity of the incident if there is a fire. No action shall be taken involving any personal risk or without suitable training.

### SPECIAL PROTECTIVE EQUIPMENT FOR FIRE-FIGHTERS:

Fire-fighters should wear appropriate protective equipment and self-contained breathing apparatus (SCBA) with a full face-piece operated in positive pressure mode. Clothing for fire-fighters (including helmets, protective boots and gloves) conforming to federal, state and local standard regulation that will provide a basic level of protection for chemical incidents.



Special Remarks on Fire Hazards: Not available. Special Remarks on Explosion Hazards: Not available.

# SECTION 6: ACCIDENTAL RELEASE MEASURES

**SMALL SPILL** : Stop leak if without risk. Move containers from spill area. Dilute with water and mop up if water-soluble. Alternatively, or if water-insoluble, absorb with an inert dry material and place in an appropriate waste disposal container. Dispose of via a licensed waste disposal contractor

**LARGE SPILL**: Stop leak if without risk. Move containers from spill area. Approach the release from upwind. Prevent entry into sewers, water courses, basements or confined areas. Wash spillages into an effluent treatment plant or proceed as follows. Contain and collect spillage with non-combustible, absorbent material e.g. sand, earth, vermiculite or diatomaceous earth and place in container for disposal according to local regulations. Dispose of via a licensed waste disposal contractor. Contaminated absorbent material may pose the same hazard as the spill product.

# SECTION 7: HANDLING AND STORAGE

**HANDLING AND STORAGE** : Store in accordance with local regulations. Store in original container protected from direct sunlight in a dry, cool and well-ventilated area, away from incompatible materials and food and drink. Keep container tightly closed and sealed until ready for use. Containers that have been opened must be carefully resealed and kept upright to prevent leakage. Do not store in unlabelled containers. Use appropriate containment to avoid environmental contamination.

**OTHER PRECAUTIONS**: Put on appropriate personal protective equipment. Do not ingest. Avoid contact with eyes, skin and clothing. Avoid breathing vapour or mist. Avoid release to the environment. Keep in the original container or an approved alternative made from a compatible material, kept tightly closed when not in use. Empty containers retain product residue and can be hazardous. Do not reuse container.

# SECTION 8: EXPOSURE CONTROLS/PERSONAL PROTECTION

**ENGINEERING CONTROLS** : No special ventilation requirements. Good general ventilation should be sufficient to control worker exposure to airborne contaminants. If this product contains ingredients with exposure limits, use process enclosures, local exhaust ventilation or other engineering controls to keep worker exposure below any recommended or statutory limits.

**RESPIRATORY PROTECTION**: Use a properly fitted, air-purifying or air-fed respirator complying with an approved standard if a risk assessment indicates this is necessary. Respirator selection must be based on known or anticipated exposure levels, the hazards of the product and the safe working limits of the selected respirator.

**EYE PROTECTION** : Safety eyewear complying with an approved standard should be used when a risk assessment indicates this is necessary to avoid exposure to liquid splashes, mists, gases or dusts.



**WORK HYGIENIC PRACTICES:** Wash hands, forearms and face thoroughly after handling chemical products, before eating, smoking and using the lavatory and at the end of the working period. Appropriate techniques should be used to remove potentially contaminated clothing. Wash contaminated clothing before reusing. Ensure that eyewash stations and safety showers are close to the workstation location.

**EXPOSURE GUIDELINES**: Emissions from ventilation or work process equipment should be checked to ensure they comply with the requirements of environmental protection legislation. In some cases, fume scrubbers, filters or engineering modifications to the process equipment will be necessary to reduce emissions to acceptable levels.

#### **SECTION 9: PHYSICAL AND CHEMICAL PROPERTIES** Physical state and appearance : Paste Odor : Characteristic Taste : Not available **Molecular Weight** : Varies Color : Grey : Not available. pН Flash Point : Not applicable Flammability (solid,gas) : Not available Melting Point : Not available. **Critical Temperature** : Not available. **Specific Gravity** :1.5 @ 20°C (Water = 1) Vapor Pressure : Not available : Not available. Vapor Density Volatility : Not available. **Odor Threshold** : Not available. Water/Oil Dist. Coeff. : Not available. Ionicity (in Water) : Not available. **Dispersion Properties** : Not available. : Insoluble in cold water and hot water Solubility Auto-ignition temperature : Not available : Not available Viscosity

# SECTION 10: STABILITY AND REACTIVITY

#### Stability Reactivity

Possibility of hazardous reactions

Incompatibility with various substances Condition to avoid Incompatible materials Corrosivity Polymerization

- : The product is stable.
- : No specific test data related to reactivity available for this product or its ingredients.
- : Hazardous reaction will not occur under normal condition and use

: Reactive with oxidizing agents.

- : No specific data
- : No specific data
- : Non-corrosive in presence of glass.
- : Will not occur.



# SECTION 11: TOXICOLOGICAL INFORMATION

### **ACUTE TOXICITY:**

This product is not classified in this hazard class. Unlike to cause harm if accidentally swallowed in small doses, though ingestion of large quantities may cause gastro-intestinal effects. **SKIN CORROSION OR IRRITATION:** 

This product is not classified in this hazard class, but prolonged or repeated skin contact sometimes may cause irritations and dermatitis.

### EYE DAMAGE OR IRRITATION:

This product is not classified in this hazard class, but direct contact may cause slight irritations. **RESPIRATORY SENSITIZATION:** 

This product is not classified in this hazard class. SKIN SENSITIZATION: This product is not classified in this hazard class. CARCINOGENICITY: Based on available data, the classification criteria are not met. **REPRODUCTIVE TOXICITY:** Based on available data, the classification criteria are not met. **ASPIRATION HAZARD:** 

This product is not classified in this hazard class.

# SECTION 12: ECOLOGICAL INFORMATION

#### Ecotoxicity BOD5 and COD

: Not available.

: Not available.

**Products of Biodegradation:** 

Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation : Not available. Special Remarks on the Products of Biodegradation :Not available.

# SECTION 13: DISPOSAL CONSIDERATIONS

### Waste Disposal:

Do not discharge in sewers, water source or tunnel. Waste must be disposed of in accordance with federal, state and local environmental control regulations.

# SECTION 14: TRANSPORT INFORMATION

DOT Classification: Not a DOT controlled material (United States). Identification: Not applicable. Special Provisions for Transport: Not applicable.

# SECTION 15: REGULATORY INFORMATION

Product information as in accordance with federal, state and local environmental control regulations.



- P.U. (A) 310/2013 Occupational Safety and Health (Classification, Labelling and Safety Data Sheets of Hazardous Chemicals)
- ACT 514 Occupational Safety and Health Act 1994, Factories and Machinery Act 1974 and all following updates
- P.U. (A) 131/2000 Occupational Safety and Health (Use and Standards of Exposure of Chemicals Hazardous to Health)
- P.U. (A) 294/2005 Regulation of the Environmental Quality (Scheduled Wastes) and all following updates
- ACT 127 of the Environmental Quality Act 1974

# SECTION 16: OTHER INFORMATION

References: Not available. Other Special Considerations: Not available. Created: 23/11/2018 15:23 PM Last Updated:

### **Desclaimer:**

The information above is believed to be accurate and represents the best information currently available to us. However, we make no warranty of merchantability or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall ScienceLab.com be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential or exemplary damages, howsoever arising.



43400, Serdang, Selangor Attention/Pegawai : EN. AHMAD HAZIQ

NU	DESCRIPTION	UNIT (KG) PRICE / UNIT (RM)	TOTAL PRICE (RM)
1	High Temperature Grease	130 215.00	27,950.00
<b>-</b>			27,950.00

### Note:

30% deposit is required for product manufacturing. Payment term in 60 days from invoice date This quotation valid within 30 days This quotation is still subject to term & conditions to be agreed upon by both parties

Thank You For Choosing Us!

Authorised by:

Dr. Nur Aainaa Syafini Binti Mohd Radzi GENERAL MANAGER

"This is a computer-generated document and it does not require a signature. This document shall not be invalidated solely on the ground that it is not signed."

UMP TE	CHNOLOGY SDN BHD	(1007461-M)	INVOICE
Lebuhraya Tu 26300 Gamba			
Pahang Darul Malaysia	Makmur	PAGE:	1 of 1
Tel: 00 540	2707 For 00 540 2000	ACCOUNT NO:	S045
Tel: 09-549 Tax Number:	2797 Fax: 09-549 3288	INVOICE NUMBER:	INV000027/18
		INVOICE DATE:	02-Nov-2018
SOLD TO:	ORCA GREENTECH PLT (SYMBIOSIS 3.0), LEVEL 1, BLOCK B,	DELIVERY NOTE NO:	
	TIC III, UPM-MTDC, UNIVERSITI PUTRA MALAYSIA SERDANG SELANGOR DARUL EHSAN U/P: ARIF BIN MD ZAIN 43400	YOUR ORDER NUMBER:	PO-180005
		TERMS:	60 days
		TRX NUMBER:	SJ 377

NO	DESCRIPTION	Quantity	Unit Price	Discount	AMOUNT (RM)
	CLAIM PAYMENT FOR PROJECT:-				
1	HIGH TEMPERATURE GREASE	130.00	215.00	0.00	27,950.00
	Terms of payment: 1. Full payment in 60 days of the invoice date				
	UMF				
RIN	GGIT MALAYSIA: Twenty Seven Thousand Nine Hundred Fifty Only		5	UBTOTAL:	27,950.00
				TAX:	0.00
				TOTAL:	27,950.00

 All check must be crossed A/C Payee Only 'NOT NEGOTIABLE' and made

 payable to:
 UMP TECHNOLOGY SDN BHD
 Affin Islamic A/C No: 105120012543

 The Company reserves the right to charge interest on overdue accounts. Immediately after

 payment you will be able to email at nikshafillah@umpholdings.my or fax no: 09-549 3288 a bank

 slip; it is a proof that your invoice have been paid

018

AUTHORISED BY

DATE



# UMP TECHNOLOGY SDN BHD (1007461-M)

Kompleks UMP Holdings, Persiaran Bandar Gambang Off Lebuhraya Tun Razak, 26300 Gambang, Kuantan, Pahang Darul Makmur Tel: 609-5493262 Faks: 609-5493288

### DELIVERY ORDER

D.O No	;	UMPT/DO/2018 (012)
Inv No	:	INV000016/18
Date	:	4/10/2018
То	;	ORCA GREENTECH PLT
		LEVEL 1, BLOCK B, UPM-MTDC TECHNOLOGY
		UNIVERSITI PUTRA MALAYSIA
		SERDANG SELANGOR
Attnt	:	AHMAD HAZIQ

NO		DESCRIPTION		QUANTI (UNIT/F		PRICE/ QTY (RM)	TOTAL (R	PRICE M)
1	G GR	EASE (HIGH PERFOR	MANCE)	30	.00	215.00	6,	450.00
		GR	AND TOTAL			<u> </u>	6,4	50.00

Thank you.

Carrier Signature

AHMAD HANAFI BIN ABU BAKAR ASSISTANT BUSINESS DEVELOPMENT

RECEIVED BY

(Receivers Signature & Stamp) 51-LGN



# UMP CONSULTANCY & TRAINING SDN BHD (644755-D)

Kompleks UMP Holdings, Perisaran Bandar Gambang Off Lebuhraya Tun Razak, 26300 Gambang, Kuantan Pahang Tel: 609-5493263/3262 Faks: 609-5493288

### Project Number: UCTQ16-058

### Full Report Covering the project activities from 28/04/2016 to 30/6/2016

Reporting Date 30/06/2016

### Chemical Testing and Analysis on Electric Contact Lubrication for Middle Voltage Switchgears

		Data Project		
Project loc	cation (	Jniversiti Malaysia Pał	nang	
Project sta	rt date: 2	28/04/2016		
Project en	d date:	30/06/2016 Extension	date:	
Procurem	ent Order 2	20784104		
Total budg	get I	RM113,000		
GST (6%)	: I	RM6,780		
(%) of elig	ible costs I	RM119,780		

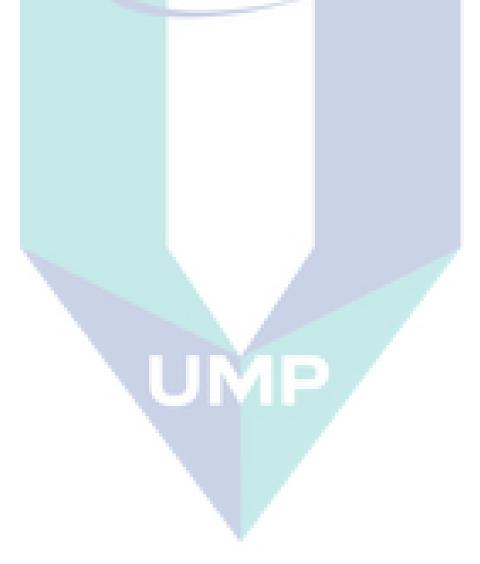
	Data Beneficiary		
Name Beneficiary	UMP CONSULTANCY AND TRAINING SDN BHD		
Contact person	MOHD SABRI BIN MAHMUD		
Postal address KOMPLEKS UMP HOLDINGS			
OFF LEBUHRAYA TUN RAZAK			
	26300 KUANTAN		
	PAHANG		
Telephone	609-5493263/3262		
Fax:	609-5493288		
E-mail	lokman@umpholdings.my		

# **Table of Contents**

1	Exe	cuti	ve summary	. Error!	Bookmark	not defined.
2	Pro	ject	Work Description	. Error!	Bookmark	not defined.
	2.1	Del	iverables of the Project*	. Error!	Bookmark	not defined.
3	Pro	blen	n Statement	. Error!	Bookmark	not defined.
	3.1	Per	formance and Physical Properties of Grease	. Error!	Bookmark	not defined.
	3.2	Che	emical Defect, Hazard and Properties	. Error!	Bookmark	not defined.
4	Met	thod	ology*	. Error!	Bookmark	not defined.
	4.1	Cor	npatibility of Contact Material*	. Error!	Bookmark	not defined.
	4.2 <b>Booki</b>		tection against Fretting Corrosion, Mechanical V k not defined.	Wear and	l Friction*	Error!
	4.3 <b>define</b>		tection against Atmospheric and Galvanic Corro	osion*	Error! Bo	okmark not
	4.4	Du	rability*	. Error!	Bookmark	not defined.
	4.5	Tes	ts of Physical Properties	. Error!	Bookmark	not defined.
	4.5.	1	Stability Test	. Error!	Bookmark	not defined.
	4.5.	2	Consistency Test	. Error!	Bookmark	not defined.
	4.5.	3	Contamination Test	. Error!	Bookmark	not defined.
	4.5.	4	Dropping point	. Error!	Bookmark	not defined.
	4.5.	5	Penetration Test	. Error!	Bookmark	not defined.
	4.5.	6	Oil bleeding	. Error!	Bookmark	not defined.
	4.5.	7	Ageing Study	. Error!	Bookmark	not defined.
	4.5.	8	Electrical Conductivity of Grease	. Error!	Bookmark	not defined.
	4.6	Tes	ts of Chemical Properties	. Error!	Bookmark	not defined.
	4.6.		Fourier Transform Infrared (FTIR) spectroscop	ру	Error! Bo	okmark not
	defi					
		okm	Thermal Gravimetric Analysis (TGA) - Changark not defined.			·
	4.6. <b>def</b> i	3 ined	Chromatographic Analysis of Grease Compone	ents	Error! Bo	okmark not
	4.6. <b>not</b>	-	Inductively Coupled Plasma Mass Spectrometr ined.	ry (ICP-N	MS) Error	! Bookmark
5	Res	ults	and Discussion	. Error!	Bookmark	not defined.
	5.1	Ana	alyses of Physical Test Results	. Error!	Bookmark	not defined.
	5.1.	1	Stability (Weight Loss) Test	. Error!	Bookmark	not defined.
	5.1.	2	Consistency and Contamination Test	. Error!	Bookmark	not defined.
	5.1.	3	Dropping Point	. Error!	Bookmark	not defined.
	5.1.	4	Penetration Test	. Error!	Bookmark	not defined.
	5.1.	5	Oil Bleeding	. Error!	Bookmark	not defined.

6 Ageing Study Error! Bookmark not defined	k not defined.
7 Electrical Conductivity of Greases Error! Bookmark not defined	k not defined.
Analysis of Chemical Test Results Error! Bookmark not defined	k not defined.
Fourier Transform Infrared (FTIR) spectroscopyError! Bookmark no ned.	Bookmark not
2 Thermal Gravimetric Analysis Error! Bookmark not defined	k not defined.
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Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Error! Bookmar	or! Bookmark
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### UMP CONSULTANCY & TRAINING SDN BHD (644755-D)

Kompleks UMP Holdings, Persiaran Bandar Gambang Off Lebuhraya Tun Razak, 26300 Gambang, Kuantan Pahang Tel: 609-5493263/3262 Faks: 609-5493288

	QUOTA	TION/ SEBUTHARGA	4	
No GST No Rujuka Client/Pel Address/A	anggan : ORCA GREEN PLT		Date/Tarikh :	4 November, 2018
Attention No Phone No Faks Email	/Pegawai : /Tel/HP :			
NU	DESCRIPTION	QTY/UNIT	PRICE/QTY (RM)	TOTAL PRICE (RM)
1	High Performance Grease From Industrial ( (G-Grease) (Technical service) *Please refer to the attachment for scope of work	Wastes L/SUM	L/SUM	30,000.00
				1,800.00
	GST (6%)			,

#### Note:

Payment term in 30 days from invoice date

This quotation valid within 60 days

This quotation is still subject to term & conditions to be agreed upon by both parties

Thank You For Choosing Us!

Authorised by:

MOHD LOKMAN CHE JUSOH BUSINESS DEVELOPMENT EXECUTIVE

"This is a computer-generated document and it does not require a signature. This document shall not be invalidated solely on the ground that it is not signed."

# **UMP CONSULTANCY & TRAINING SDN BHD**

(644755-D)

INVOICE

	·····	
Malaysia	ACCOUNT NO:	TDU2093
Tel: 09-549 2794 Fax: 09-549 3288	INVOICE NUMBER:	UCT001226/18
Tax No: C10-1808-31025916	INVOICE DATE;	23-Nov-2018
	DELIVERY NOTE NO:	UCT1801115
SOLD TO: ORCA GREENTECH PLT	YOUR ORDER NUMBER:	
SYMBIOSIS 3.0, LVL 1, BLOCK B, UPM-MTDC TIC III,	TERMS:	30 days
UNIVERSITI PUTRA MALAYSIA	SALESPERSON:	
SERDANG SELANGOR DARUL EHSAN	TRX RATE (\$):	1.0000
43400	TRX NUMBER:	SJ 8777
NO	sc. AmeBEITax	A Amount((\$)
PROJECT NO       UCT1801115         CLAIM PAYMENT FOR PROJECT:- AHAMD         HAZIQ BIN AHMAD         1       HIGH PERFOMANCE GREASE FROM INDUSTRIAL WASTE (G-GREASE)- (CONSULTANCY)       1.00       30,000.00	0.00 30,000.00 6% 1,80	00.00 31,800.00
RINGGIT MALAYSIA: Thirty One Thousand Eight Hundred Only	TOTAL BEFORE SERVICE TOTAL	TAX: 1,800.00

All check must be crossed A/C Payee Only 'NOT NEGOTIABLE' and made (Affin Islamic A/C No: 105120010736) payable to: UMP CONSULTANCY & TRAINING SDN BHD The Company reserves the right to charge interest on overdue accounts. Immediately after payment you will be able to email nikshafillah@umpholdings.my or fax no: 09-549 3288 a bank slip; it is a proof that your invoice have been paid.

AUTHORISED İΒΥ

DATE

E&O.E

### **REPORT OF CONSULTANCY**

# TITLE : HIGH PERFORMANCE GREASE FROM INDUSTRIAL WASTE (G-GREASE)

CLIENT : ORCA GREEN PLT

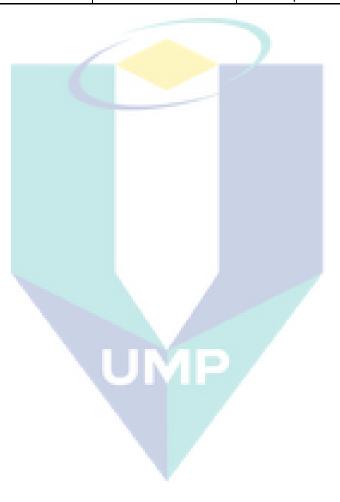
No	Activities	Scope of work	Deliverables	Actual Delivered	Date of Delivered	% of Fees and Values
1	Education on Product Detail and Presentation Assistance	<ul> <li>1.1 To provide Orca GreenTech PLT the product literature and relevant information in conducting technology and marketing validation activity. These are the list of documents needed :</li> <li>Journal relating to grease</li> <li>-MSDS</li> <li>-IP Registration Front Cover</li> <li>- Certificate of Analysis (COA-ASTM)</li> <li>- Report on performance &amp; specification based on COA-ASTM</li> <li>-SOP for production(general)</li> <li>-etc</li> <li>UMP give information in form of reading material via email or post to Orca GreenTech PLT. The source of the literature must be reliable and proven facts.</li> </ul>	-Received notes -Related documents and articles -Reports -Lists of SOPs	-Refer to email 1.1.1 Requesting information 10.4.2017.pdf -Refer to email 1.1.2 – Dokumen Untuk CRDF 1.pdf	-10 <sup>th</sup> April 2017 -31 <sup>st</sup> May 2017	10% RM3180.00

		1.2 UMP to prov ide explanation in details about the product/technologies by discussion. Discussion will be held from time to time or once related problem is faced and can be done by meeting or through phone call. UMP will train members of the Orca GreenTech PLT what to understand about the product/technologies and able to re-explain to others. UMP to provide simple analogies to educate people who are not familiar with the technology and product. A question and answer sessions between UMP and Orca GreenTech regarding the product.	- Discussion /meeting request by Orca -Minute of Meeting (MOM) -Slides -Flow Chart/Diagram	-Refer to Minute of Meeting 1.2.1 M.O.M Orca 7 MAC 2017.pdf -Refer to email 1.2.2 – Info untuk Commercialization Plan.pdf -Refer to email 1.2.3 – CRDF 1 Application_Additional information_clarification required by Dr Raihana.pdf	-7 March 2017 -2 <sup>nd</sup> June 2017 -13 <sup>th</sup> August 2017	15% RM4770.00
2	Product Testing Assistance	To guide Orca GreenTech PLT in types of product testing and analysis required for grease and suggests where to conduct the test. UMP will provide lists of parameter for each testing. UMP will assists through the testing procedure and to pass all the parameters for the following tests : -Physical Testing (ASTM) -Chemical Testing UMP will discuss with Orca GreenTech on the feedback received from the testing and ways to improve the product.	- Discussion /meeting request by Orca -Types of test required -List of parameter for each test -Testing procedures	-Refer to email 1.1.2 – Dokumen Untuk CRDF 1.pdf - Refer to email 1.2.3 – CRDF 1 Application_Additional information_clarification required by Dr Raihana.pdf -Refer to email 2.1 Grease testing Enquiries 31 May 2018.pdf	-31 <sup>st</sup> May 2017 -13 <sup>th</sup> August 2017 -31 <sup>st</sup> May 2018	15% RM4770.00

3	Product	3.1 UMP to supply Orca GreenTech	-UMP to deliver	-Refer to email 1.1.2 –	-31 <sup>st</sup> May 2017	10%
	Supply and	product	product to Orca	Dokumen Untuk CRDF 1.pdf		RM3180.00
	Improvement	that have been manufactured and the	GreenTech PLT.	-Refer to email 1.2.3 – CRDF 1	-13 <sup>th</sup> August 2017	
	Support	quality is tested according to SOP. UMP	(list of SOP and	Application_Additional		
		will also be handling the packaging and	QC)	information_clarification		
		the labeling according to SOP. UMP will	-Manufacturing	required by Dr Raihana.pdf		
		deliver the product to Orca GreenTech	SOP by UMP	-Refer to email 3.1.1 –	-4 <sup>th</sup> April 2018	
		upon the order made.		Requesting for a new G-grease		
				with temperature		
				specification.pdf		
				-Refer to document 3.1.2	-19 <sup>th</sup> July 2018	
				Grease sticker 19 July 2018.pdf		
				-Refer to diagram Refer to	-19 <sup>th</sup> July 2018	
				diagram 3.1.3 Grease		
				Packaging.jpg		
				-Refer to diagram 3.1.4 Label	-21 <sup>st</sup> August 2018	
				Grease.png		
				-Refer to 3.1.5 Delivery Order	-4 <sup>th</sup> October 2018	
				Grease ORCA 30kg.pdf		
		3.2 Based on feedback from technology	- Discussion	-Refer to email 3.2.1 –	-7 <sup>th</sup> October 2018	15%
		and	/meeting request	Perbincangan Projek CRDF 1		RM4770.00
		marketing validation, UMP and Orca	by Orca	Symbiosis-MTDC Bersama Orca		
		GreenTech will have a discussion on	- Continuous	GreenTech Sdn Bhd.pdf		
		product improvement including	Quality	-Refer to 3.2.2 Minit Mesyuarat	-17 <sup>th</sup> October 2018	
		packaging and labeling. UMP to also	Improvement	Bersama ORCA GreenTech		
		guide and advise on technical	(CQI) by UMP	-Refer to slide 3.2.3 Proposal		
		improvement		Grease Capacity	-30 <sup>th</sup> October 2018	
				30.10.2018.pptx		
			Ŧ	-Refer to 3.2.4 Minit Mesyuarat		
				Bersama ORCA GreenTech &	-30 <sup>th</sup> October 2018	
				MTDC		

				-Refer to 3.2.5 Email Feedback Form Review.pdf -Refer to 3.2.6 G-Grease Feedback Form.pdf	-31 <sup>st</sup> October 2018 -31 <sup>st</sup> October 2018	
4	Quality Control	UMP to carry out quality control procedures of the product at the facility of the appointed manufacturer according to SOP to ensure sample product meets the specifications, safe to use and merchantable quality along the process of validation of technology and market.	- Discussion /meeting request by Orca -UMP provide SOP for Quality Control and reports	-Refer to SOP 4.1 Double Jacketed Mixing Tank.pdf -Refer to SOP 4.2 Cone Penetration.pdf -Refer to SOP 4.3 Consistency Test.pdf -Refer to SOP 4.4 Contamination Test.pdf -Refer to SOP 4.5 Dropping Point Analysis.pdf -Refer to SOP 4.6 Mechanical Grease Worker.pdf -Refer to SOP 4.7 Oil Bleeding Test.pdf -Refer to email 4.8 – COA dan MSDS untuk G-Grease.pdf - Refer to 4.9 SDS LCG 015 (Version 1).pdf	-21 <sup>st</sup> August 2018 -11 <sup>th</sup> September 2018 -11 <sup>th</sup> September 2018 -11 <sup>th</sup> September 2018 -11 <sup>th</sup> September 2018 -11 <sup>th</sup> September 2018 -11 <sup>th</sup> September 2018 -12 <sup>th</sup> September 2018 -12 <sup>th</sup> September 2018 -12 <sup>th</sup> September 2018 -3 <sup>th</sup> November 2018	15% RM4770.00
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### AUSTRALIAN JOURNAL OF BASIC AND APPLIED SCIENCES

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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, solium 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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#### Mohd Najib Razali et al, 2017

#### Australian Journal of Basic and Applied Sciences, 11(3) Special 2017, Pages: 154-159

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°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  $\frac{1}{2}$  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  $\frac{1}{2}$  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.

#### Mohd Najib Razali et al, 2017

#### Australian Journal of Basic and Applied Sciences, 11(3) Special 2017, Pages: 154-159

#### 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  $40\mu m$  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.

Funed silica serves as a universal thickening agent and an anticaking agent (free-flow agent) in powders. Funed 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).

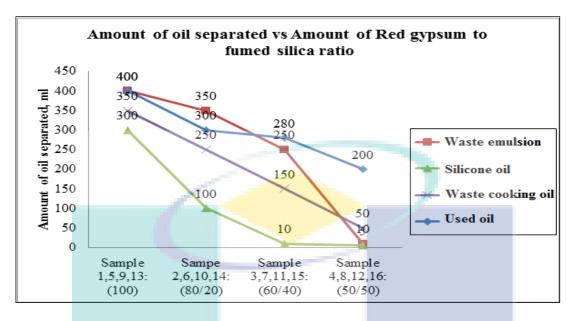
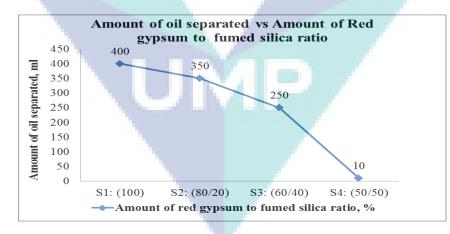


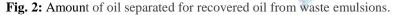
Fig. 1: Grease stability graph by four types of base oil.

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.





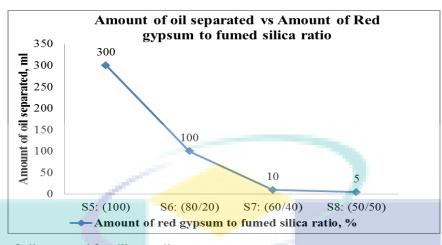


Fig. 3: Amount of oil separated for silicone oil.

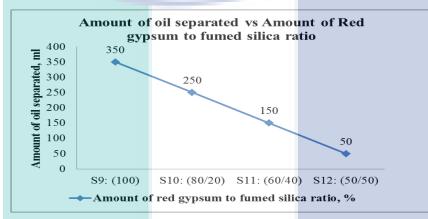
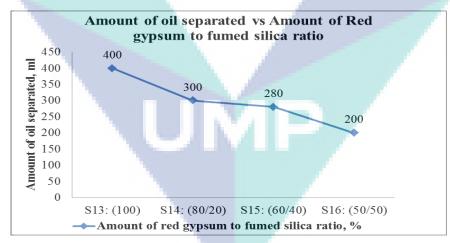


Fig. 4: Amount of oil separated for waste cooking oil.





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.

#### Mohd Najib Razali et al, 2017

#### Australian Journal of Basic and Applied Sciences, 11(3) Special 2017, Pages: 154-159

#### **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. Funed 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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# Formulation of lubricating grease using Beeswax thickener

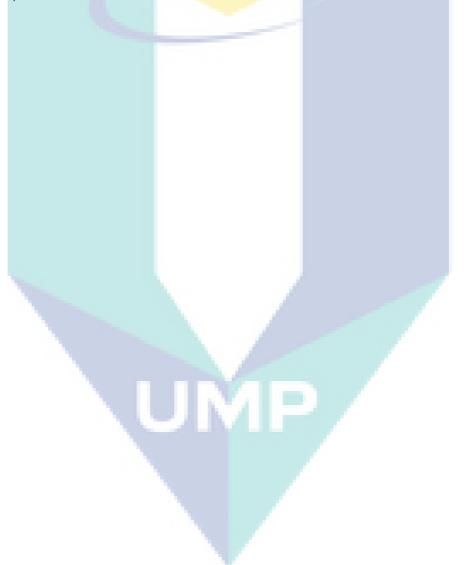
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## Formulation of lubricating grease using Beeswax thickener

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Abstract. The issues on environmental pollution has brought the industries to seek the alternative green solutions for lubricating grease formulation. The significant challenges in producing modified grease are in which considering the chosen thickener as one of the environmental friendly material. The main purposes of the current research were to formulate lubricant grease using different types of base oils and to study the effect of thickener on the formulated lubricant grease. Used oil and motor oil were used as the base oils for the grease preparation. Beeswax and Damar were used as thickener and additive. The grease is tested based on its consistency, stability and oil bleeding. The prepared greases achieved grease consistency of grade 2 and 3 except for grease with unfiltered used oil. Grease formulated with used oil and synthetic oil tend to harden and loss its lubricating ability under high temperature compared to motor oil' grease. Grease modification using environmental friendly thickener were successfully formulated but it is considered as a low temperature grease as the beeswax have low melting point of 62°C-65°C.

#### 1. Introduction

Most of the lubricants used nowadays lead to environmental pollution [1]. The environmental legislation by government bodies as well as higher consumer awareness are influencing the design process of the lubricating grease to employ more sustainable and environmentally friendly components [2]. Due to this scenario, a new market revolving around eco-friendly products is developing very rapidly, where consumers exhibits higher tendency to use new products and is also willing to pay for green products to conserve the environment from negative effects [3]. Several studies has been done related to the environmental friendly lubricating grease through the utilization of eco-friendly thickener such as cellulose derivatives and beeswax [4,5].

Grease is a semi-fluid product which comprises of a thickening agent along with other elements that provide the grease with special properties dispersed in a lubricating base oil. The common percentage compositions for base oils, thickeners and additives are 70-95%, 3-30% and 0-10% respectively [6]. Many studies has been done related to eco-friendly grease, where most researchers utilized vegetable oil as the base oil [7–9]. In contrast, the amount of studies done on thickeners are scarce. Thickeners are known to play a vital role in grease composition for which it determines the consistency of grease.

Used oils are generally generated during the engines service and maintenance however, it is often contaminated with impurities in the course of usage and handling. In addition, used oils often points out as a serious pollution problems due to irresponsible act of illegal dumping and unmanageable disposal

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resulting in groundwater, surface water and soil contamination[10]. Thus, in this study, used oils were utilized as one of the base oils to prevent the aforementioned problems.

The thickener's main function is to remain in contact with or hold lubrication from moving or leaking out under the force of gravity and often referred to as a sponge that holds the lubricants [11]. Beeswax is a natural substance obtained from the honeycombs of bees after the honey has been removed by draining or centrifuging. It possesses characteristic of insoluble in water, sparingly soluble in alcohol and very soluble in ether. In this study, beeswax was used as the thickeners in grease composition. Damar (*Shorea dipterocarpaceae*) are solid resins, generally hard and durable, were used as grease additive. It provides a unique depth of gloss, superior color and gloss retention, adhesion and flexibility [12], and it has melting point ranging from 85°C-115°C.

Lubricating grease functions in reducing the wear and tear of the surfaces by avoiding direct metal to metal contact between the rubbing surfaces and lowering the expansion of metal due to frictional heat and destruction of material. Motor oils and used oils, beeswax as well as damar were used to produce grease. This research study was carried out to formulate grease using different types of base oils and to study the effect of thickener on the formulated grease.

### 2. Methodology

#### 2.1. Materials

Different types of base oils were used which are motor oils and used oils. The grade of the motor oils chosen were Toyota Premium Mineral (PM), Toyota Synthetic Formulation (SF) and Perodua Semi Synthetic (SS). Used oil collected from workshop was selected to be another alternative of base oils. Beeswax was chosen to act as a thickener, is a natural substance obtained from the honeycombs of bees after the honey has been removed by the process of draining or centrifuging meanwhile the additive, Damar (*Shorea dipterocarpaceae*) a solid resin is the sap from naturally wounds to the surface of the tree.

## 2.2. Preparation of grease samples

The compositions to produce 200g of grease samples were fixed with ratio of 14:5:1 of base oil, thickener and additive, respectively. 50g of beeswax was slurred together with half of the amount of base oil (70g) in a 500-mL beaker at continuous stirring rate. The speed of the mixer was adjusted according to the viscosity of the paste and for each of the sample, the mixing was continued until a smooth paste was formed at a constant temperature of 50°C-60°C. The other half amount of base oil and Damar (10g) were added slowly into the paste and the process continuous until all the materials blend together. The samples for each formulated grease were labelled as follows in accordance to its base oils types:

1	
Type of base oil	Sample identification
Toyota Premium Mineral	PM
Toyota Synthetic Formulation	SF
Perodua Semi-Synthetic	SS
Used oil (unfiltered)	WO
Used oil (filtered)	FWO

#### Table 1. Grease samples identification.

#### 2.3. Consistency test

The tests on each sample were performed using the SKF Grease Test Kit TGKT 1. A fixed grease volume was spread between the two glass plates for 15 seconds by means of the weight. The consistency of the grease strain was observed and evaluated using calibrated measuring scale NLGI grade. This test method

was in accordance of ISO 2137 which specifies methods for determining the consistency of lubricating greases when only small samples were available. The results of NLGI for each grease indicates their consistency level [13].

NLGI Number	Worked Penetration, tenth of millimeter	Consistency
000	445-475	Very soft
00 0	400-430 355-385	Soft
1	310-340	Soft
2 3	265-295 220-250	Creamy texture (buttery) Semi-solid
4	175-205	Stiff
5	130-160	Stiff
6	85-115	Hard solid

<b>Table 2.</b> NLGI classification system based on consistency.
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### 2.4. Stability test

The test was done as complied with the Standard Test Method for Oil Separation from Lubricating Grease during Storage (ASTM-D1742). It is known as oil separation test in which it determines the stability of the grease formulated. All grease samples were stored in enclosed container and the initial level of the grease is observed and recorded. The samples were left for 2 months at room temperature. The amount of oil separated was measured using the measuring cylinder and weighed. Theoretically, the lesser the oil separated, the better the stability or quality of the grease. The sample is considered stable if and only if the amount of the oil separated less than 4% [14].

#### 2.5. Oil bleeding test

The tests on each sample were performed using the SKF Grease Test Kit TGKT 1. A fixed amount of grease was put on a piece of blotter paper provided. The samples were heated for two hours at constant temperature of 60°C according to the recommended operating condition of the grease test kit [15]. The base oil released from the grease created a stained on the paper. The bleed area and the percentage difference between bleed area of fresh and used samples were calculated by using equation (1) and (2).

$$S... = 0.785 \times (D_{AV...}^2 - 100)$$
(1)

$$\%\text{Diff} = 100 \times \frac{(\text{S}_{\text{Used}} - \text{S}_{\text{Fresh}})}{\text{S}_{\text{Fresh}}}$$
(2)

### 3. Results and discussion

### 3.1. Modified grease appearances

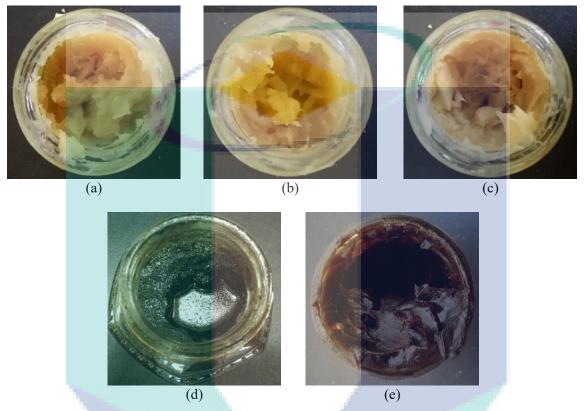


Figure 1. The appearances of modified grease samples. (a) Toyota premium mineral-oil-based grease, (b) Toyota synthetic-oil-based grease, (c) Perodua semi-synthetic-oil-based grease, (d) unfiltered used-oil-based grease.

The modified grease samples prepared were semi-solid and their color depend on the color of base oil which vary from light yellow to black as in Figure 1. The properties of modified greases are as in the following subtopics.

### 3.2. Formulated lubricant grease consistency

The outcomes from the testing done upon each of the grease samples in accordance to the NLGI consistency grade (Table 2). The grades defined the consistency of the greases and their appearance at 25°C. SF-based grease, SS-based grease, and FWO-based grease were recorded NLGI grade 2 and PM-based grease of grade 3, where grease with these consistencies were most commonly used in general industrial, automotive and others [16]. Grease with NLGI Grade 2 were suitable for rolling bearings moderately loaded with medium speed applications. Grease of this class of consistency are formulated to give a good balance of properties required for easy pumping through dispensing systems [17]. In addition, most of the multipurpose grease also of this grade of NLGI 2-3 consistency.

	5 1 1	e
Sample	NLGI	Consistency
PM	3	Firm
SF	2	Buttery
SS	2	Buttery
WO	00	Semifluid
FWO	2	Buttery

 Table 3. Consistency of prepared modified grease.

Comparing the WO-based grease and the other samples, very large difference in consistency observed for WO-based grease of which it is of NLGI grade 00. WO-based grease is softer than the other prepared greases. By comparing WO-based grease and FWO-based grease, FWO-based grease is formulated with filtered used oil. The presence of contaminants in the unfiltered used oil (WO) might be affecting the grease consistency for which most of these contaminants is removed before being used in FWO-based grease formulation [18]. Therefore, it was inferred that the WO-based grease consistency can be increase by decreasing the oil-to-thickener ratio [19].

#### *3.3. Oil separation during storage*

Greases tends to release their base oils during storage (static bleeding). Static oil bleeding can be affected by storage conditions and is more pronounced if the grease is soft in consistency (NLGI grades 00, 0 and 1) [20]. Oil separation often relates to the base oil and thickener combination with kinematic viscosity of the base oil [21]. All grease samples were stored at room temperature for 2 months and were observed for any changes of the behavior and properties. Table 4 shows oil separation only occurred to WO-based grease.

Table 4. Grease oil sepa	ration upon storage	at 25°C.
Oil separated (g)	%loss	Description
-	/	Stable
-	-	Stable
-		Stable
16.20	8.10	Unstable
-		Stable
	Oil separated (g) - - -	

WO-based grease was found to be unstable and recorded excessive oil separation which is more than 4%. Contaminants present in the unfiltered used oil can draw out the base oils from the thickener system over time [20]. However, the oil puddle on top of the grease does not result in the grease being unsuitable for use. It can be either removed by decanting the free oil from the surface or by manually stirring it back into the grease.

Prepared grease other than WO-based grease shown to have very good oil separation resistance. From the appearance of the other grease samples, the base oil and the thickening agent chosen seems to have a strong physically-acting bond forces that build a stable three-dimensional network structure [22]. Interestingly, filtered used oil-base grease (FWO) has good oil separation comparable to the fresh motor oils-base grease. This is might be due to the pre-treatment of used oil that was done such as settling, filtration and dehydration for which most of the contaminants in the used oil were being removed.

#### *3.4. Oil bleeding of the formulated grease*

This test method is not suitable for greases softer than NLGI grade 1 consistency due to the tendency for softer greases to sweep away while the test is performed. Therefore, the testing was not applicable on WO-based grease of grade 00. Comparison of grease bled between fresh samples and used samples grease were made. bled area difference (%Diff) between the used and the fresh sample were calculated

to indicated either the lubricating ability of the grease. Positive values mean the grease lubricating ability is increased or vice versa.

Based on the results in table 5, PM-based grease and SS-based grease shows the positive value in the percentage of area different which indicates that the grease lubricating ability can be maintained at high temperature. Other greases indicated reduction on their lubricating ability. SF-based grease and FWO-based grease losses its lubricating ability when subjected to high temperature for which it causes these greases harden as time goes. The rate of oil released from the grease will increase with time and vary based on the temperature at which it is stored [23]. It was observed that the formulated greases have low working temperature range which was found to have similarity with Kreivaitis' [5] study, as he stipulated that the dropping-point temperature of the beeswax-thickened greases were within the range of 52°C to 66°C. However, the formulated grease high temperature tolerance can be improved by adding proper additive to increase its dropping point temperature properties.

Table 5. Oil bleeding test at 60°C.

			-		
Samp	ble D <sub>AV fresh</sub> (mm	) $D_{AV used}$ (mm	n) $S_{\text{Fresh}} (\text{mm}^2/\text{s})$	s) $S_{\text{Used}} (\text{mm}^2/\text{s})$	%Diff
PM	34.0	36.5	828.96	967.31	16.68
SF	45.0	43.0	1511.13	1372.97	-9.14
SS	40.0	42.0	1177.50	1306.24	10.93
WO	N/A	N/A	N/A	N/A	N/A
FWO	35.5	34.0	910.79	828.96	-8.98

Table 6 shows the data analyzed from the tests carried out on each of the grease samples. The tests carried out includes the consistency test, stability test and oil bleeding test. Consistency test were done to determine the NLGI consistency grade for grease samples. In stability test, any changes in grease samples were analyzed after 2 months of storage at room temperature. Oil bleeding tests were carried out for two hours at constant temperature of 60°C where the base oil released from the grease of fresh and used samples created oil stained on the paper. Both the diameter of the stained and the bleeding properties were measured.

Sample	Color	Appearance	NLGI	Stability @ 25°C	%Diff oil bleed @ 25°C
PM	Light yellow	Firm	3	Stable	16.68
SF	Light yellow	Buttery	2	Stable	-9.14
SS	Light yellow	Buttery	2	Stable	10.93
WO	Black	Semifluid	00	Unstable with 8% oil separated	Not applicable
FWO	Dark brown	Buttery	2	Stable	-8.98

Table 6. Overall results of formulated lubricant grease analysis.

## 4. Conclusion

Beeswax-thickened greases are potentially an environmental friendly type of grease. Based on the result obtained, most of the formulated grease were successfully formulated by utilizing beeswax as thickener with NLGI consistency range of grade 2 - 3. However, these greases are not meant to be subjected to high temperature as it tends to lose it lubricating ability and harden as it exposed to high temperature for a period of time.

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Research paper



## **Grease and its Application on Electrical Equipment: a Review**

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#### Abstract

Grease hardening and dry-out have been part of the major challenges in grease usage in electrical industry. The findings obtained over the years related to the study of synthetic, specialty, and new grease have found that the usage of these greases are costly; hence, it is very necessary to find an alternative method to reduce the cost as much as possible. Increasing petroleum demands, depletion of petroleum reserves, and the environmental awareness have influenced a huge interest in the use of waste oil as the alternative of base oil for grease formulation. The waste oils are considered as a promising candidate due the fact that the re-refining process of base oil is relatively cheap with high yield and the recovery of good quality oil. Optimum grease formulation is necessary in solving the aforementioned issues as well as overcoming complication specifically in the electrical industry where these greases are mainly utilized.

Keywords: Electrical equipment; Grease application; Grease formulation; Lubricating grease; Waste oil.

## 1. Introduction

Greases are semisolid lubricants that are utilized to reduce friction between two surfaces [1] as well as a protective film which allows the two touching surfaces to be separated. According to the American Society for Testing and Materials (ASTM), lubricating grease is defined as "a solid or semi-fluid lubricant consisting of a thickening agent in a liquid lubricant. Other ingredients imparting special properties may be included". Greases are commonly made of 85 to 90 % base oil and 10 to 15% thickener. Hence, both the thickener fibers and the oil will resist deformation when stress is applied to grease, which respectively opposes the elastic and viscous forces [2].

Thickeners are dispersed in greases to produce a gel-like structure due to the fact that greases are made of lubricating oils. Lubricating grease does not flow and it shares the properties of solid at normal temperature, thus they are capable of maintaining their shape. However, the gel structure of grease will break down and flow normally like lubricating oils when the load is applied and the grease is heated. Most interestingly, grease is featured with reversibility that helps the flow of grease to stop and allows it to return to solid form when the load is removed [3]. However, lubricating greases will lose their solid properties when the load and temperature are applied to a critical value.

According to Watson [4], the first use of grease dates back to about 1400 B.C. and it was first introduced by the Ancient Romans and Egyptians. These greases were made from animal fats, resins, and lime. However, industrial development has come a long way since then which consequently led to the introduction of lithium, barium, and aluminum soap base greases in the 1920s and 1930s. The progressive industrial development has led to the establishment of complex base greases in the 1940s and 1950s. Therefore, complex base greases are slowly replacing the conventional greases due to its properties which include high temperature performance and good performance at moderate temperature.

The demand for environmental friendly products has always been a major point in any commercial sales. On top of that, both environmental legislation and consumer awareness have been impacting the design of lubricating grease. The original environmental friendly lubricant developed in the 1970s and 1980s was made to be applied on chainsaw and outboard motor applications [5]. A number of researches related to bio-lubricant grease have been performed by utilizing biodegradable raw material such as vegetable oil and base oil [6–8]. However, the development of biolubricant grease formulation was observed to be costly compared to the current grease available in the market; hence, it is considered to be impractical [9].

According to Lugt [10], advanced material technologies such as the implementation of nanotubes were also explored in the last decade. However, these types of technology do not exhibit any technological breakthrough in terms of functional performance apart from being commercially unpromising until the present time. Most of the existing researchers have focused on the development of grease performance or efficiency [11–13]. Nevertheless, there is a wide gap in the area of grease formulation related to the main components of grease that is largely made of crude oil (85%-95%) with recycled materials or waste products. The formulation process conducted using the downstream petroleum raw material is very expensive plus the permanent declining of petroleum resources.

Donley [14] further suggested that greases are formulated to perform the following key functions: (i) keep moving part apart, (ii) reduce friction, (iii) transfer heat (iv) carry away contaminants, (v) transmit power, (vi) protection, and (vii) corrosion prevention. Generally, grease can be categorized into several types based on its potential applications which include clay greases, extremepressure (EP) greases, soap-based grease, multi-purpose greases, and others. Lubricating grease has been widely utilized for numerous applications involving machinery and moving part, in which it functions as a sealant, provides protection against corrosion, and at the same time reduces noise and shock.



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#### 2. Page layout

It is extremely crucial to note that lubricating grease and oil share no similarities. Grease is usually utilized when it is more practical to be used instead of lubricating oil. In this case, the type of lubricant used for a particular application is determined based on the equipment design and working conditions [15]. In general, lubricating grease is largely used in the heavy industry related to automotive, steel, and machinery as well as light industry involving food and textile.

#### 2.1. Automotive Industry

For many years, conventional automotive greases were made from either aluminum, calcium, or sodium soaps based on the requirements dictated by the operating condition [16]. Take one case whereby EP greases that were utilized in automotive wheel bearing, chassis, disc brake, universal joints, and fifth wheel are either in the state of lithium or lithium complex, aluminum complex, and calcium sulfonate greases [17].

Meanwhile, lubricating grease selected in automotive industry should possess the following important properties: high dropping point, good shear stability and adhesive properties, thermal and oxidation degradation resistance, water resistance, good protection against heavy and continuous shock loads, anti-wear, good compatibility with other conventional greases and differential oils [18, 19]. On top of that, these properties are required to ensure that the grease is able to perform well during the operation. Lubricating greases of National Lubricating Grease Institute (NLGI) Consistency Grade 2 are often used in the automotive application due to their ability of remaining soft in cold weather.

#### 2.2. Steel industry

In the case of steel industry, various types of greases were applied to different parts of machineries. The steel plant equipment is able to work under different operating parameters which include extreme heavy load to high speed as well as low to high temperatures [20]. Therefore, it is very crucial to properly select the most appropriate grease to ensure smooth operation and reliability of equipment. The greases used in steel industry are not particularly similar to those of automotive industry as they have high load-carrying capacity, oxidation stability, mechanical stability, water wash resistance and good pumpability, and low cost for large-volume consumption [21]. In most cases, the grease is used to lubricate machinery with the operating temperature of less than 135 °C [19]. Other than that, EP multi-purpose greases that are comprised of lithium and aluminum complex soaps are normally utilized in steel mills.

Apart from that, the steel industry also adopts a wide variety of bearings that are lubricated with grease such as plain journal bearings which is especially beneficial and useful in rolling mill operations, rolling-element bearings, and table and roll neck bearings. On the other hand, the type of grease that are based on mineral oil used in hot and cold rolling equipment include conventional EP lithium greases, calcium complex greases, calcium sulfonate complex greases, lithium complex greases, and aluminum complex greases [16]. Finally, it should be duly noted that greases applied to these bearings will only be applied during the maintenance instead of the operation.

#### 2.3. Food Processing

In the case of food processing, it is necessary for food-grade greases to comply with food, and health and safety regulations [10]. Hence, the common base oils that are suitable for this particular industry include white oils, polyalphaolefin (PAOs), some esters and vegetable oils [20, 22]. On top of that, the common approved thickeners are aluminum stearate, aluminum complex, organoclay, and polyurea.

#### 2.4. Textile

The utilization of lubricating grease in the textile industry is for the purpose of lubricating the textile machinery. It should be acknowledged that this industry prefers grease that are easier to clean and does not leave any stain to the textiles or fabrics. A number of grease products suitable for this industry usually contain additives that are able to assist in the process of removing oil stains from the fabric [20]. The common properties of greases used in the textile industry include load-carrying capacity, oxidation resistance, and good adhesion. Additionally, greases with rust preventive properties are also used due to the humid environment at the industry.

#### 2.5. Electrical Equipment

The reliability of electric utility equipments such as connectors, switchgears, and circuit breakers are possible to be improved with modern lubrication technology and practices [23]. In recent years, various types of synthetic lubricants have demonstrated the ability to outperform conventional lubricants at extreme temperatures, resistance ability of grease dry out, and ability to protect against wear and corrosion which can lead to the failure of the equipment [24]. Electrical equipment installed in both industrial and commercial facilities can be exposed to both indoor and outdoor environments which are potentially detrimental to the metals used in electrical contacts including their coatings; therefore, it is very crucial to choose the most suitable lubricants to ensure the equipment is well protected.

In addition, lubricants are able to improve the performance of electrical contact by either reducing mating forces, extending the durability of plating, and enhancing the protection against corrosion [25]. However, the general principle of electrical equipment lubrication revolves around protecting the electrical connection from degrading which can further increase the electrical resistance [26]. Moreover, deposits on the contact surfaces formed by corrosion will definitely lead to the increase in contact resistant. Meanwhile, the application of lubricants in electrical contacts seems to provide protection from harsh environment, galvanic/fretting corrosion, and wear/friction, without interfering with the contacts' electrical resistance.

A wide range of lubricants have been tested on electrical contacts [25, 27, 28]. In the same token, Polyphenyl Ether (PPE) and Perfluorinated Polyether (PFPE) were discovered to possess high-temperature withstanding properties in comparison to other types of lubricants. Additionally, PPEs are also capable of providing protection from corrosion as well as increasing the life of connectors apart from having good thermal stability [29].

There are relatively few historical studies in the area of lubricating grease application in electrical equipment compared to other field of applications. Most researchers put their focus on the process of formulating grease with excellent conductivity. However, a number of problems were found related to the use of lubricating grease in electrical equipment such as long-term stability and high operating temperature. Most greases tend to evaporate at the temperatures over 100  $^{\circ}$ C [26] which then leads to the formulation of lubricating grease with excellent performance at extreme temperature.

### 3. Issue of Grease Usage in Electrical Industry

The application of lubricants is one of the ways to prevent any potential factors that can possibly increase the electrical resistance involving an electrical connection. The type and function of the contact determine the specific type of the required lubricant. In light of this, electrical contact can be defined as two surfaces that are in contact by force with the passage of electric current, voltage, or signals. The vast majority of published articles or journals on electrical lubrication have been observed to offer two strong recommendations [30] which are described as follow: (1) follow manufacturer's instruction, and (2) avoid mixing the lubricants. Apart from that, several OEM recommendations also include engineering specifications, maintenance book, and documentation. The issues related to the need of following the guideline revolve around the difficulty to access the original manual that is included together with majority of hardware.

On top of that, there are several problems faced by the electrical industry related to the use of grease. In relation to this, it can be further described based on the issues faced by one of the electrical power industry in Malaysia which is related to their switchgears that were made of copper with or without silver plating. The switchgear often faces mechanical failure and electrical overloads at peak hours, which then leads to the elevation of temperature that will indirectly affect the grease. Apart from that, it was also observed that grease tends to experience major challenges in electrical contact lubrication which are dried-out and hardening. Grease dry-out and hardening are majorly the result of temperature effect during operation. Both low and high operating temperature can lead to grease hardening, but it is a different case for low temperature which is highly dependent on the type of base oil used in the formulation [15].

Moreover, there are several possible causes that can lead to the dry out and hardening of grease with respect to temperature effect. Low viscosity base oil used in grease tends to have high risk of volatilization at high temperature due to the evaporation of oil from the matrix over time, which further results in the ratio of oilto-thickener to drop and to the hardening of grease [31]. Another cause of grease hardening can be explained by the oxidation of grease over time. It should be understood that both base oil and thickener in grease tend to have the possibilities to oxidize, but thickener is less prone to oxidation compared to base oil [15].

The buildup of oxidation products can be deleterious to thickener structure as well as cause the bleeding of oil particularly when grease is oxidized. High operating temperature can cause the oxidation rate to accelerate, which then cause carbonization that will definitely result in the progressive grease hardening and crust formation [32]. Not only that, it can also cause the increase of contact resistance which then reduces the contact reliability. Additionally, over-greasing can lead to oxidation since churning of grease may occur which finally results in energy loss and rising temperatures [33].

A research conducted by one of Malaysia's' electrical power industry team had managed to discover that grease applied to the contact area is capable of changing color (blueish green slurry and polymer-like) after a period of time. Generally, the discoloration of grease on the components generally indicates that oxidation is taking place , thus leading to the increase of contact resistance [34]. Another interesting finding is that lubricants may degrade as it reacts with the atmosphere and likely to polymerize when heated in the presence of a copper-based alloy [35]. Moreover, copper is known to degrade polymers through catalytic-enhanced oxidation; hence, it will definitely cause grease to change if it gets in contact with copper at elevated temperature [36] which will also induce copper corrosion. Therefore, it is important to utilize grease with a copper corrosion inhibitor as it may reduce lubricant-induced copper corrosion [37].

Nevertheless, several established studies have pointed out that grease hardening and dry-out can be avoided. Grease applied on contact surfaces are exposed to four temperature sensitive aging mechanisms, which are evaporation, surface migration, polymerization, and degradation [30, 38]. However, lubricants may degrade by oxidation as well as form insulating films and increase contact resistance with time and temperature [39]. Therefore, it is extremely crucial to select proper grease formulation by referring to the electrical equipment design and operating conditions in order to avoid equipment or contact failure. Other than that, material properties such as viscosity, thermal stability, surface tension and vapour pressure are very important to be considered in order to select lubricating grease for electrical contact application [30].

Numerous types of greases were applied and tested by the team based on the greases recommended by manufacturers (OEM) for the purpose of electrical contact lubrication. However, the current practices seem to face several complications compared to normal operation due to the shortage of stocks. Hence, this further increases the need to acquire greases from different OEMs which can lead to the utilization of non-recommended contact grease. Along these lines, it can be safe to infer that not all kinds of greases are suitable for electrical contact application.

The contact between two metal surfaces is highly reliant on boundary lubrication. Hence, a very thin film of lubricant or additive, or both must be maintained to ensure that boundary lubrication is effective. This can be achieved by using various types of additive such as extreme pressure, anti-wear, and lubricity additives. Apart from that, solid lubricants, such as graphite, molybdenum disulfide, and PTFE may also be added by the lubricant manufacturer. However, both PTFE and molybdenum disulfide seem to have the possibility to increase in wear [40].

#### **4. Electrical Contact Grease**

The lubrication of separable electronic connector can be described as the practice of coating contact surfaces with the purpose of reducing mechanical wear and friction as well as degradation caused by fretting. Meanwhile, lubricants are described as thin metallic film, fluids, greases, and solid. In relation to that, this review has chosen to focus on the use of grease to lubricate the electrical contact surfaces. Grease is defined as a threedimensional (3D) matrix soap fibers dispersed throughout a liquid lubricant. In other words, it can be considered similar to a sponge soaked with liquid that will flow as a result of applying shear stress exceeding the yield point. The base oil is bonded by the following factors: (1) capillary forces, (2) mechanical occlusion, or (3) molecular attraction between polar region on soap fibers and base oil molecules [41].

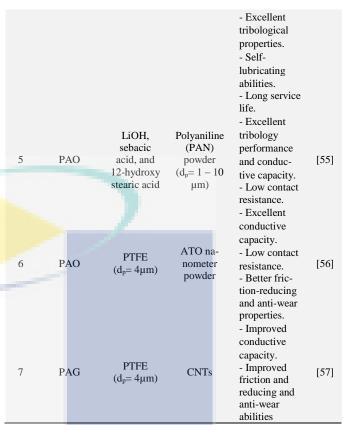
The contact surfaces were not flat in contact operation even when force is applied, and the two surfaces do not touch at all points [42]. Grease lubricating mechanisms can be described as oil reservoir that is slowly released (bleeding) into the contact surface. However, some believe that the oil is released by the destruction of thickener fibers instead of bleeding. In contact surface lubrication, the initial filling is believed to play a major role [43]. However, over-greasing may eventually lead to excessive churning, elevation of temperature, and severe grease degradation. This finding is constant to the finding of Zhang [44] which states that the appropriate thickness of lubrication film is the most influential factor on inhibition from corrosion and wear, which is also capable of reducing the possibility of contact failure.

According to Campbell [45], grease viscosity is limited to the speed of slide and the lowest temperature in service. In this case, petroleum-based grease is believed to work effectively provided that the bearing component is fully rotated continuously at a specified speed. However, it stays static in circuit breaker, while partial rotation and movement tend to occur during operation [46]. In addition, petroleum-based grease does not perform well in extreme temperature and dirty environment. Hence, the grease will end up breaking down and becoming acidic, which then forms a layer of varnish on the contact surface. In line with Slade's statement [47], mineral oil is often expected to protect contact surface from tarnishing but the opposite tends to occur over time. Mineral oil is mostly derived from the refining of crude oil (petroleum), and even a highly refined mineral oil is known to contain sulphur compound which is extremely corrosive. Therefore, plating the metal contact surface can slow down the build-up of resistive film in mineral oil environment. A research carried out by Arnell and Andersson [48] had found that the low contact resistance on copper plated with silver contact in mineral oil can be maintained by applying solid lubricant to the contact.

Furthermore, electrical contact resistance which is caused by electrical constriction tends to reduce the volume of material used, whereas film resistance tends to increase the contact resistance due to the presence of contamination film. The contamination film

that is known to be mechanically brittle may be caused by the formation of oxides or corrosion product on contact surface, which has the possibility to increase contact resistance and reduce contact reliability. However, electrical contact can be established when the film is fractured and metal-to-metal contact spot is formed by metal extrusion through the layer's crack [47]. Electrical constriction can be reduced by applying more load due to the fact that contact resistance is inversely exponential to load applied. In regard to this, the practice of contact lubrication is as old as connectors themselves [28]. The capability of lubricants to stabilize contact resistance, reduce wear, and delay the oxidation rate by protecting the metal surface from the environment is apparently not dependent on the lubricants composition and viscosity [49]. Several lubricants such as polyphenyl ethers were discovered to be more potent in inhibiting adhesive wear. However, mineral oil and polyolester are not potent to reduce the friction coefficient as a result of lower viscosity [25], including the earlier statement that describes the potential of mineral oil in causing corrosion on contact surfaces. Moreover, greases formulated using mineral oil are more prone to evaporation compared to synthetic greases, and they tend to separate from thickeners over time. However, the cost of synthetic grease is expensive compared to the conventional grease. The results provided by established studies related to the electrical contact grease formulation are displayed in Table 1. It can be observed that many contact greases possess beneficial effect on the contact resistance behavior. A vast majority of the studies show that the grease application helps in reducing the contact resistance. In spite of that, previous research on contact grease mainly focused on base oil and thickeners, and less on additive. On top of that, most of the current researchers put their concern on the use of different type of compositions of the grease in order to prevent wear and friction, along with the importance of providing excellent conductivity capacity and reducing contact resistance. However, lubrication was confirmed to only be able to delay fretting corrosion instead of totally prevent it from happening [50].

Та	ble 1: Lubricati	on test for vario	ous grease type	es in electrical con	tact
No.	Base oil	Thickener	Additive	Findings	Ref.
1	PFPE	2.8% fumed silica	-	<ul> <li>Unhindered current flow.</li> <li>No variation in passage when applied to electrical switch con-</li> </ul>	[51]
2	PFPE (M <sub>w</sub> = 2600 - 12500 gmol <sup>-1</sup> )	PTFE (d <sub>p</sub> = <1µm)		tact. - Less $M_w$ : oil evaporate and hardened. - More $M_w$ : grease too viscous. - $d_p$ = <1 $\mu$ m: contact not damage. - $d_p$ = >1 $\mu$ m: cohesion and adhesion is induced and increase thickness of lubricant film.	[52]
3	Ionic liq- uids (ILs) Lithium ILs	30% PTFE (d <sub>p</sub> = 4μm)	-	<ul> <li>Conductive grease.</li> <li>Better friction-reducing properties.</li> <li>Anti-wear at room temperature and high temperature (150 °C).</li> <li>High con-</li> </ul>	[53]
4	(Li(PEG)X)	17% PTFE	-	ductivity.	[54]



\*PFPE–Perfluoropolyether; PAO–Polyalphaolefin; PTFE– Polytetrafluoroethylene; CNT–Carbon Nanotube; ATO–Antimony Tin Oxide

### 5. Grease Formulation for Electrical Equipment

In this review, two main factors have been considered for the purpose of formulating electrical contact grease, which are the cost and problem related to the electrical industry. As has been previously stated, there exists a huge gap in the area of grease formulation. The formulation process that utilizes the downstream petroleum raw material is deemed to be expensive. In addition, the use of petroleum product in lubricant has been observed to generate several drawbacks to the contact surfaces. On top of that, it was found that synthetic grease, specialty grease, and new base oil developed for electrical equipment are more expensive compared to the traditional grease; hence, it is necessary to find an alternative method to cut down the grease production cost.

#### 5.1. Base Oil: From Crude Oil to Waste Oil

The formulation of lubricating grease commonly involves the use of petroleum or synthetic mineral oil as the base oil, but the importance of acknowledging environmental awareness has led to the establishment of more strict policies on the use of nonenvironmental friendly sources. In relation to this, the development and application of environmental friendly lubricating grease have started to gain attention but it is undeniable that eco-friendly base oil such as biodegradable synthetics ester is deemed costly. Therefore, the continuous search for the best and suitable ecofriendly product that can replace mineral oil in various industrial applications is very important, particularly in the fuel and energy sector. Hence, renewable resources such as vegetable oil and their derivatives are considered as the potential replacements for mineral oil base stock in certain lubrication application, especially in the formulation of lubricating grease [60].

The recent crude oil production has been experiencing a massive decline worldwide. In Asia-Pacific, the decline was observed to be substantially greater compared to the production pullbacks in other parts of the world [61]. In this, case, it is forecasted that the Asia-Pacific crude oil production will fall by 1 million barrels per day by 2020 which is caused by the decline of Asia-Pacific major oil producers, namely China, Indonesia, Malaysia, and Thailand.

Apart from that, vegetable oil is also well known for its biodegradability. Most of the commercial production of vegetable oil mainly revolves around for food purposes. Vegetable oils have been considered as a result of their advantages which include high viscosity index, high lubricity, and low volatility among other oils [7]. However, it is not recommended to utilize this source in the formulation of grease because it can definitely pose a negative effect on the food market. In addition, a huge number of researchers have tried to develop vegetable-oil-based derivatives which possess a nearly similar properties and performance to the petroleumbased diesel oil. However, the process involves a very high manufacturing cost. Following this, waste cooking oil was then claimed to be the suitable alternative for vegetable oils. However, it is no longer recommended to be used in food industries due to toxic build up after being heated as well as the change of oil physical properties [62, 63].

The used lubricating oil or waste oil is normally generated from the crankcase of internal combustion engines (mainly run on petrol or diesel). Apart from that, used oil is also produced and collected from other operating equipment such as hydraulic oil or gear and transmission oils [64]. However, lubricating oil becomes unfit for further utilization as a result of the accumulation of contaminants in the oil as well as the chemical changes in the oil. On top of that, the disposal, dumping, and uncontrolled burning of used oil can harmfully threaten the environment by causing severe pollution. According to the European Environmental Policy [65] on the Directive for the disposal of waste oil, it can be said that the regeneration of waste oil (producing base oils) is highly encouraged instead of the usual disposal in order to reduce the disposal amount of oil which indirectly keep the environment safe [66]. Audibert [67] states that the re-refining of oil is able to produce one-to-one ratio of new oil production which further suggests that the production of re-refined oil is also able to reduce the number of new oil produced. A study conducted by Mensah-Brown [68] shows that a total of 73.3% oil is recovered from the re-refining process of used oil, which agrees with the fact that oil can be recycled to yield lube stock to produce lubricants and sustain crude petroleum.

All in all, the study on recycled used oil, high cost of grease production, and issues related to the environmental conservation have brought a wide and promising interest in the use of waste oil as the best potential replacement for base oil in electrical contact grease production as well as possible methods that can be applied to cut the cost. It is further believed that the use of waste oil in grease formulation will be more economic because the re-refining process is relatively cheap compared to virgin base oil stock [68]. Apart from that, it also helps to prevent unreasonable dumping of used oil which can definitely save the environment from being extremely polluted.

#### 5.1.1 Waste Oil Treatment

The conventional method on the re-refining technologies for waste oil requires the vacuum distillation process which concentrates the contaminants in the vacuum residue. It is important to note that the conventional acid/clay re-refining process has been utilized for quite a number of years, but the process is not eco-friendly due to the large production of pollutant. This method is also incapable to re-refine the modern lubricating oil such as multi-grade engine oils [69, 70]. In relation to this, many acid/clay re-refining plant were shut down in 1980s [71] as a result of the increased environmental awareness, which subsequently lead to the development of improved re-refining technologies for both environmental and product quality reasons.

Furthermore, the study on the regeneration of used oil continues to develop with better emphasis on ensuring that the processes are environmentally friendly. The research performed by Hamawand [70] shows that glacial acetic acid was used to replace sulfuric acid in the method of acid/clay re-refining. This method is considered suitable because it involves a lower cost process as a result of using low-cost acid as well as moderate conditions of the process. On top of that, this process does not emit poisonous gases and pose minimum negative impact on the processing equipment.

In regard to this, the solvent extraction technique was later developed to replace the acid treatment. The basic of this pretreatment can be described based on the use of a solvent to selectively extract the base oil components from the used oil in a process that is regarded to be quite similar to the commonly used process adopted in crude oil refining for the purpose of separating asphaltenes [69, 70, 72, 73]. Generally, ketones and alcohols have been widely used as solvents, while the oil generated from this process is as good as to that produced through the acid/clay method. However, this method is costly due to the need of utilizing expensive solvents and vacuum distillation. Recently, hydrocarbon such as propane has been used as solvent [70, 71, 74] due to its ability to dissolve paraffinic or waxy material and intermediately dissolve oxygenated material.

As stated by Dang [69], the existing re-refining processes involve three basic steps which are settling, solvent chemical treatment or vacuum distillation, and finishing by hydrotreating or clay contact. Several variations and combination of processes have been developed for the purpose of lube re-refining over a period of time such as the Kinetics Technology International (KTI) process, which is a combination of vacuum distillation and hydro-finishing [75]. This technology is able to produce high quality base oils with a yield of approximately 82 % as well as to minimize the pollution cause by the by-products.

#### 5.2. Thickeners

The function of thickeners is to provide a physical matrix that can hold the base fluid in a solid structure until the operating conditions such as load, shear, and temperature are able to initiate viscoelastic flow in the grease. Hence, a careful balance of solubility between the base fluid and the thickener is required in order to achieve this matrix. In other word, thickener is an agent that is capable of changing the phase from fluid to semi solid.

The two principal groups of thickeners can be divided into two: metal soaps non-metal soap. Soap-based greases are by far the most widespread lubricants because metal soap consists of a longchain fatty acid neutralized by metal such as lithium, sodium, calcium, aluminum, and others. Meanwhile, inorganic, organic, and synthetic materials are used as thickeners in non-soap greases. Nevertheless, non-soap grease has managed to gain popularity in high-temperature environments. However, under elevated temperatures, base oil will eventually oxidize even though the thickeners are able to withstand high temperature, which requires a frequent relubrication interval [15].

#### 5.3. Additives

The purpose of adding additives to some grease is to improve their lubricating properties. Moreover, additive also plays an important role in protecting grease and lubricated surfaces apart from its ability to enhance the grease performance. Additives may constitute 0 to 15 percent of the total grease mass; hence, it is added to increase additional properties such as anti-oxidation, rust prevention, anti-wear, and pressure resistance. A wide range of additives can be used to improve grease performance, whereby molybdenum disulphide (MoS<sub>2</sub>) have gained interest in this review because it has high load-carrying capacity and excellent in both low and high temperature[15].

The use of  $MoS_2$  is very beneficial in the boundary lubrication regime based on the presence of metal to metal contact, which is contrasting to the hydrodynamic regime where the presence of sufficiently thick fluid film is able to prevent asperity contact and any virtual wear [76].  $MoS_2$  can be described as an anisotropic transition metal dichalcogenide (TMD) compound material with a trigonal prismatic structure that is comprised of a layer of molybdenum atoms between two layers of sulphur atoms that are loosely bounded by Van der Waals interaction [77].

The electrical properties of  $MoS_2$  are of significant practical importance, particularly in electrical application. Various parameters are able to potentially correlate with  $MoS_2$  resistivity, but both the temperature and level of impurities are considered to be of a higher significant [78]. On top of that, there is no clear agreement on electrical conductivity of  $MoS_2$ , but a strong correlation seem to exist between  $MoS_2$  conductivity and temperature [79]. In relation to this,  $MoS_2$  electrical conductivity tend to increase with temperature as the resistivity decreases [78]. In short, it is safe to conclude that  $MoS_2$  is very versatile to be used as additive in the formulation of electrical contact grease due to its high temperature withstanding properties and good electrical conductivity.

### 6. Conclusion

In general, it has been observed that research on grease for electrical equipment is still ongoing and most of the current researchers tend to focus on the optimization of grease performance or efficiency. In addition, it is fully noted that the current development of grease for electrical equipment which include synthetic, specialty, or new finding is deemed to be very expensive. Moreover, the study on waste oil regeneration has been conducted since the 1930s due to the environmental issues caused by the waste oil. The present review further covers grease application on electrical equipment, including grease production performed using waste oil for electrical contact. Most of the challenges in this field are related to the need of finding the optimum formulations that is very necessary in producing lubricating grease which can be applied for specific type of electrical contact. Finally, the advancement of lubricating grease which utilizes used oil can lead to major revolution in the world of lubricant market because this idea is believed to be effective in reducing the cost and environmental impact related to grease manufacturing.

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#### Egyptian Journal of Petroleum xxx (xxxx) xxx



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## Full Length Article

# Formulation of fumed silica grease from waste transformer oil as base oil

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#### ABSTRACT

This study aimed to formulate fumed silica (FS) grease using waste transformer oil (WTO) and to investigate the grease properties. Various unexplored application of the reused WTO due to its reusable characteristic. WTO and fresh transformer oil (ITO) were used as grease base fluid and the properties of oil and grease were evaluated using ASTM International standards and SKF's Grease Test Kit. The oil and grease chemical compound were determined using Fourier Transform-Infrared Spectroscopy at wavenumber of 500–4000 cm<sup>-1</sup>. It was found that WTO have high viscosity index of 96 and low moisture content of 0.05% after pre-treatment than ITO. FS greases with and without MoS<sub>2</sub> were classified as NLGI 2 greases when FS content was 8%. Grease formulated with MoS<sub>2</sub> shows better performances in term of corrosiveness, oil bleed within -15% to +15%, oil separation less than 4%, and dropping point of >300 °C. FTIR results shows no significant different between ITO and WTO, and between all formulated greases. Based on the findings, it was concluded that WTO can be utilized as an alternative base oil in grease formulation due to the good properties exhibited by the formulated WTO-based FS grease blended with MoS<sub>2</sub>.

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#### 1. Introduction

Grease is known as a semisolid product that consists of base fluid, thickener, and additive. Nowadays, there are variety types of grease existed. The rapid growth of the industries has led to higher demands in lubrication as well as increasing amount of waste oil generated. Waste oil is categorized as schedule waste and is known for its ability to threaten public health and environment.

Green product had gained its attention in the recent years. In Malaysia, waste oils is generated approximately 150 million liters annually [1] and an estimated 36 MTA of waste transformer oil (WTO). The idea of reusing waste oil to produce new product has been implemented in Malaysia [2] but unfortunately, WTO has not making its way to be reused in any application. Even though there are only small amount of WTO disposed every year compared to waste engine oils but, it is important to find the potential use of WTO in order to avoid the amount to increase.

Grease consist of almost 90% of base oil which is mainly derived from petroleum oil. However, the amount of petroleum oil is continuously decreasing due to the limited petroleum reserves. Researchers been studying other alternative raw materials which have the potential to use as a replacement of the petroleum oil

Peer review under responsibility of Egyptian Petroleum Research Institute. *E-mail address:* suhailajapar@live.com (N.S. A. Japar). such as vegetable oil, waste cooking oil, waste engine oil, ionic liquid and more [3-6].

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Transformer oil is an insulating oil which act as heat transfer medium in the transformer. In general, transformer oil is produced from wax-free naphthenic oils. After certain period of being used in the transformer, the oil is removed and form a waste known as WTO. Several studies on the utilization of transformer oil in grease formulation have been conducted by blending the oil into lubricating oil and by using transformer oil solely as base oil to study the potential of the transformer oil to provide dielectric and insulating properties for the greases, respectively [7–9]. However, there are no study conducted regarding the use of WTO oil in the formulation of grease.

Earlier findings of testing the possibility of grease structure to form when WTO is utilized in the formulation, it was found that the grease structure is comparable to the preferable grease texture at specific formulation. Based on that, the current study aimed to investigate the potential of WTO as a grease's base oil substitute and to evaluate the formulated greases structure and properties.

#### 2. Experimental work

#### 2.1. Materials

Waste transformer oil (WTO) collected from electrical power plant located in Selangor, a fresh uninhibited industrial grade

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2

N.S. A. Japar et al. / Egyptian Journal of Petroleum xxx (xxxx) xxx

2.3.1. Consistency test

transformer oil (ITO), fumed silica (FS) powder, molybdenum disulfide ( $MoS_2$ ) with 98.5% purity.

#### 2.2. Grease formulation

#### 2.2.1. Pre-treatment of WTO

In this experiment, WTO was pre-treated through several treatments to remove unwanted contaminants such as water and suspended solid. WTO was first settled in an untouched container for a week at room temperature to separate the WTO and water layers by gravity forces. Water layer separated at the bottom of the container was then removed, leaving only the WTO inside the container. WTO was then transferred into new cleaned enclosed container.

Secondly, WTO was vacuum filtered at room temperature using vacuum pump and glass microfiber filter with pore size of  $1.2 \,\mu$ m to remove any visible solid particles suspended in the WTO. The WTO was heated on a hotplate at 120 °C for at least 2 h with continuous stirring to remove moisture and any volatile compound present in the WTO through evaporation process. After evaporation process, WTO was cooled down to room temperature before being stored for further use.

#### 2.2.2. Preparation of grease containing ITO (IG<sub>i</sub>)

Grease containing ITO was formulated to compare its properties with grease formulated using WTO. There were two formulations involved, for which the greases were formulated with and without the addition of additive which was MoS<sub>2</sub>.

ITO was heated to 80–90 °C and FS was added portion wise for 30 min with continuous homogenization and  $MoS_2$  was added afterwards. Homogenization was continued for a total time of 1 h to disperse the thickener and additive [10]. After homogenization, grease was stored in enclosed container for 2 days to allow cooling of grease. The formulation of grease containing industrial transformer oil (IG<sub>i</sub>) was shown in Table 1.

#### 2.2.3. Preparation of grease using WTO (WG<sub>i</sub>)

This experimental grease formulation consisted primarily of WTO and FS. There were two formulations involved, for which the greases were formulated with and without the addition of additive which was MoS<sub>2</sub>.

WTO was heated to 80-90 °C and FS was added portion wise for 30 min with continuous homogenization and  $MoS_2$  was added afterwards. Homogenization was continued for a total time of 1 h to disperse the thickener and additive [10]. After homogenization, grease was stored in enclosed container for 2 days to allow cooling of grease. The formulation of grease containing waste transformer oil (WG<sub>i</sub>) was shown in Table 1.

#### 2.3. Grease analysis

All formulated greases were analyzed through analytical testing to determine their properties.

#### Table 1

Formulation of the greases IG<sub>i</sub> and WG<sub>i</sub>.

#### Components (%wt) Sample notation $IG_1$ $IG_2$ $WG_1$ $WG_2$ $WG_3$ $WG_4$ WG<sub>5</sub> $WG_6$ WG<sub>7</sub> $WG_8$ Industrial transformer oil 92 91.14 Waste transformer oil 95 93 92 91 93.1 91.14 90.16 89.18 Fumed silica 8 6.86 5 7 8 9 7.84 8.82 4.9 6.86 Molybdenum disulfide 2 \_ \_ 2 2 2 2

The test on each grease was performed using the SKF Grease Test Kit TGKT 1. A fixed grease volume was spread between the two glass plates and pressed for 15 seconds by means of the weight. The consistency of the grease strain was observed and evaluated using calibrated measuring scale NLGI grade. This test method was in accordance of ISO 2137 which specifies methods for determining the consistency of lubricating greases when only small samples were available. The results of NLGI for each grease indicates their consistency level [11].

#### 2.3.2. Dropping point test

The dropping point test was conducted as described in standard ASTM D2265 (Standard Test Method for Dropping Point of Lubricating Grease over a Wide Temperature Range) [12]. In this test, grease sample was placed in grease test cup supported in a test tube and placed in an aluminum block oven at a preset constant temperature. A sample thermometer is placed in the tube and so positioned that it measures the temperature. As the temperature increases, at some point a drop of material will fall from the cup to the bottom of the test tube. The reading on the sample thermometer is recorded to the nearest degree as the observed dropping point. At the same time, the temperature of the aluminum block oven is also recorded to the nearest degree. The drop point was then calculated using Eq. (1).

$$DP(^{\circ}C) = ODP + [(BT - ODP)/3]$$
<sup>(1)</sup>

where, DP stands for dropping point, ODP is a thermometer reading when first drop reaches the bottom of the test tube, and BT is the block oven temperature when the drop falls.

#### 2.3.3. Oil bleeding

This method was in accordance to IP 121 where large volume of sample was required [13,14]. Oil bleeding test was conducted using the SKF Grease Test Kit TGKT 1, an alternative method where only small volume of samples were required [15]. The sample was put on blotter paper and heated for 2 h at 60 °C according to the SKF's manual [13]. The oil stain created on the paper were measured based on the bleed area and the percentage difference between bleed area of fresh and used samples by using equation Eqs. (2) and (3). Used greases refer to grease that have been aged at two controlled condition of 10 days at room condition and at 70 °C [16].

$$S_i = 0.785 \times (D_{AV...}^2 - 100)$$
 (2)

% Diff = 
$$100 \times \frac{(S_{Used} - S_{Fresh})}{S_{Fresh}}$$
 (3)

where,  $S_i$  stands for the bled are from fresh and used sample,  $D_{AV}$  is the average diameter of the bled area, and %Diff represents the bled area difference between fresh and used sample.

#### 2.3.4. Oil separation

Oil separation test was carried to identify the tendency of oil to be separated from grease during storage in accordance to ASTM

## **ARTICLE IN PRESS**

N.S. A. Japar et al. / Egyptian Journal of Petroleum xxx (xxxx) xxx

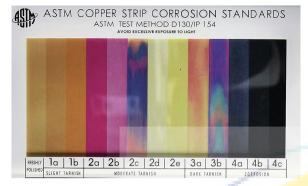


Fig. 1. ASTM copper strip corrosion standards.

D-1742 (Standard Test Method for Oil Separation from Lubricating Grease during Storage). The grease sample was stored in an enclosed container at room temperature for 1 month. The quantity of oil separated was collected and measured in weight percentage.

#### 2.3.5. Corrosion test

Corrosion test was carried out to determine the level of corrosiveness of the base oil and the formulated grease towards copper strips. This method was carried according to ASTM D-130 (Standard Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test) and ASTM D-4048 (Test Method for Detection of Copper Corrosion from Lubricating Grease by the Copper Strip Tarnish Test), respectively [17,18]. In this test, a prepared copper strip is totally immersed in a sample of grease or oil and heated in an oven or liquid bath at a specified temperature for a definite period of time. The condition for the test was set at 100 °C for 3 h in water bath for base oil and 24 h in an oven for grease. At the end of the test period, the strips were removed from sample, washed and compared to the ASTM Copper Strip Corrosion Standards (Fig. 1) [18].

#### 2.3.6. FTIR analysis

Fourier transform infrared (FTIR) spectroscopy identifies the type of base oil and thickener in grease sample. By comparing the fresh grease reference to the used grease, FTIR spectrum can provide information regarding contamination and any changes in a grease sample. In this experiment, greases and oils were characterized at wave number from 500 to 4000 cm<sup>-1</sup>.

#### 3. Result and discussion

#### 3.1. Physicochemical properties of oil

Table 2 shows the physicochemical properties of ITO and WTO. ITO's properties was as in data sheet provided by the industry. Base oils are present in majority of >80% in greases formulation where it functions to take care of lubrication by seeping out from the thick-ener matrix after being introduce to load [19]. High viscosity oil-based greases flow slowly compared to low viscosity oil-based greases. Wherever application speeds are low, high viscosity oil-based greases are recommended and vice versa. The choice of oil viscosity depends on the intended application. It is clear that in this study the transformer oils possessed lower viscosity of 10 cSt at 40 °C.

#### 3.2. FTIR characterization of oil

FTIR had been used to determine the functional group of ITO and WTO. The measurement was within 500–4000 cm<sup>-1</sup>. Figs. 2

#### Table 2

Physicochemical properties of ITO and WTO.

Properties	Test	Industrial transformer oil	Waste transformer oil
Appearances Kinematic viscosity	Visual ASTM D445	Clear & bright	Bright Yellow
at 40 °C, cSt		9.84	10.20
at 100 °C, cSt Viscosity index	ASTM D2270	2.55 81	2.61 81
Density, g/mL	Gas pycnometer	0.895	0.875
Moisture content, %	Karl Fischer method	0.002	0.05

and 3 shows that the oils have high intensity bands in the region of  $2852-2950 \text{ cm}^{-1}$ , as a result of the CH<sub>2</sub> and CH<sub>3</sub> asymmetric stretching. A strong band at 1455 cm<sup>-1</sup> due to the C–H asymmetric bending vibrations which indicates the presence of alkanes [20]. Moreover, a strong band obtained at 1376 cm<sup>-1</sup> also indicate the functional group of alkane C–H bond stretch of CH<sub>3</sub> bond [21]. A weak band at 721 cm<sup>-1</sup> represented the C–H out of plane bend indicating the presence of alkanes [22].

By comparing the FTIR spectrums of both ITO and WTO, there was no significant differences between fresh and waste transformer oil. In addition, there was no clear peaks indicating the presence of contaminants or by-products especially in WTO, such as dissolved gases, moisture etc. for which it usually resulted from the thermal stress in the transformer [21]. Thus, there were no further WTO treatments conducted to remove any dissolved contaminants other than the pre-treatment of oil.

#### 3.3. Consistency of formulated grease

Table 4, shows the test results of formulated greases on its consistency, dropping point, oil bleeding and the oil separation.

The outcomes from the consistency test done upon each of the grease samples is in accordance to the NLGI consistency grade (Table 3). The grades defined the consistency of the greases and their appearance at 25 °C. Based on Table 4, IG<sub>1</sub>, IG<sub>2</sub>, WG<sub>3</sub>, WG<sub>4</sub>, WG<sub>7</sub>, and WG<sub>8</sub> were recorded with NLGI grade 2 to NLGI grade 3, where grease with these consistencies were most commonly used in the industries [23]. Greases with NLGI grade 2 were suitable for rolling bearings moderately loaded with medium speed applications. Grease of this class of consistency are formulated to give a good balance of properties required for easy pumping

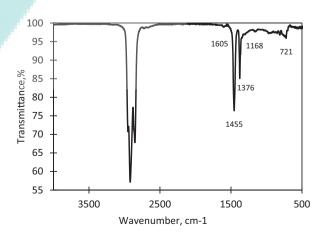
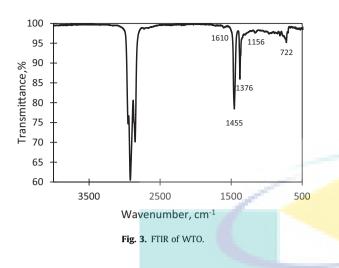


Fig. 2. FTIR of ITO.

## ARTICLE IN PRESS

N.S. A. Japar et al. / Egyptian Journal of Petroleum xxx (xxxx) xxx



through dispensing systems [24]. In addition, most of the multipurpose grease also of this grade of NLGI 2-3 consistency.

Some of the formulated greases possessed NLGI grade consistency softer than NLGI 2 for which it might be due to the insufficient amount of thickener in the formulation to hold the oil in the thickener system. However, these soft greases consistency can be increase by decreasing the oil-to-thickener ratio [25]. Conversely, greases with softer consistency does not mean it cannot be used in any application. Greases of such consistencies can used at low operating temperatures and high-speed application. Therefore, the grease consistency may be adjusted in the formulations according to the application's operating condition.

#### 3.4. Dropping point of grease

Table 4 shows the results of dropping point test for the formulated greases using both ITO and WTO. Based on the results, the formulated greases dropping point temperature increases as the

#### Table 3

NLGI classification system based on consistency [23].

NLGI grade	Worked penetration, tenth of millimeter	Consistency
000	445-475	Very soft
00	400-430	
0	355-385	Soft
1	310-340	Soft
2	265–295	Creamy texture (buttery)
3	220-250	Semi-solid
4	175–205	Stiff
5	130–160	Stiff
6	85-115	Hard solid

#### Table 4

Formulated grease properties.

amount of FS increased in the formulation. Furthermore, the addition of additive into the formulation also affecting the grease's dropping point, as MoS<sub>2</sub> was known for its better performance under both low and high temperature [26,27]. FS and MoS<sub>2</sub> is known for their high melting point of 1600 °C and 1700 °C, respectively. These results are in agreement with the previous studies by Abdulbari et al. [10] and Mohd Najib et al. [28], where there was no dropping point exist for grease formulated using FS even though the temperature exceed 350 °C and 240 °C, respectively. Therefore, the high dropping point of the formulated greases show that the greases are thermally stable and have good heat resistance over high temperature.

#### 3.5. Oil bleeding of grease

Oil bleeding properties of grease refers to the tendency of grease to bleed oil. A certain amount of bleeding is considered desirable in greases since this tends to provide continuous oil lubrication to the applied area. Bleeding properties usually will depend on the grease's thickener structure, base oil viscosity and grease firmness. In this test, oil bleeding properties of grease was conducted by evaluating the oil bleeding difference between oil bleeding area of fresh grease and aged grease in both room condition and at 70 °C. According to Bots [29], when the bleeding difference is within plus or minus 15 percent (+15% > X > -15%), the grease still can be used without changing the re-lubrication intervals.

By referring to Table 4, the grease formulated without additive shows less oil bleeding at 70 °C compared to at the room condition for which, this indicates that the greases were already begun to dry out at this temperature. Lugt [30] stated that when the oil bleeding shows that there is 50% oil left in grease composition, the change in grease type and re-lubrication interval is required. Interestingly, the addition of MoS<sub>2</sub> in all grease formulation shows significant improvement in term of oil bleeding difference at both room condition and 70 °C. This indicates that MoS<sub>2</sub> were able to prolonged the grease lifetime. These results were in agreement with Hangzhou [31] where he found that MoS<sub>2</sub> could delay the process of grease deterioration. Similar to Bhardwaj et al. [32] which stipulated that the grease blended with MoS<sub>2</sub> shows lesser damage to thickener structure and lead to long grease life.

#### 3.6. Oil separation from grease

After storage of 1 month, all grease samples were observed for oil separation on the surface of grease. The amount of oil separated from grease was shown in Table 4. According to ASTM standard [33], grease of NLGI less than 1 is not suitable for the standard test method. However, when the test was conducted, it was shown that the greases of consistency NLGI 000 were the only grease with oil separation, as expected due to their soft consistency. However, the

Grease sample C	Consistency (NLGI grade)	Dropping point (°C)	Oil bleeding (%)		Oil separation (%)
			Room condition	At 70 °C	
IG <sub>1</sub>	2-3	>350	-7.75	-14.2	No separation
$IG_2$	2-3	>350	-8.39	-11.4	No separation
WG <sub>1</sub>	000	203.3	Not tested (too soft)		0.14
WG <sub>2</sub>	1-0	312.3	-11.01	-28.7	No separation
WG <sub>3</sub>	2	>350	-7.87	-15.4	No separation
WG <sub>4</sub>	3-4	>350	-28.98	-28.40	No separation
WG <sub>5</sub>	000	270.7	Not tested (too soft)		0.17
WG <sub>6</sub>	1	>350	-7.08	-14.8	No separation
WG <sub>7</sub>	2	>350	-0.28	-11.7	No separation
WG <sub>8</sub>	3-4	>350	-1.16	-15.3	No separation

amount of oil separated from the grease was within the acceptable value of less than 4% [30]. FS function as thickener and tends to hold and attract the oil in its thickener system.  $WG_1$  and  $WG_5$  only contained 5% amount of FS for which apparently, it was not enough to hold 95% of oil. Insufficient amount of FS in the formulation results in the formation of oil layer on the grease surface [28].

#### 3.7. Detection of copper corrosion from grease

Tables 5 and 6 show the corrosiveness results of the base oil and the formulated grease with and without additive. In this study, it was found that the ITO and WTO were only slightly tarnish towards copper strips at which the appearances were close to the freshly polished strips (Fig. 4). The corrosivity of the formulated grease using WTO with and without additive were similar (Classification 1 - slight tarnish). However, greases formulated using ITO seems to have different corrosivity level of classification 1 (IG<sub>2</sub>) and classification 2 (IG<sub>1</sub>). IG<sub>1</sub> was formulated using ITO without MoS<sub>2</sub>. When compared to WG<sub>1-4</sub>, FS might not responsible for the grease corrosivity, which is in lined with results obtained by Abdulbari et al. [10]. Aside from that, the addition of MoS<sub>2</sub> in grease formulated using ITO (IG<sub>2</sub>) was found only slightly tarnish towards the copper strips. This viewpoint is not in line with other findings where the addition of MoS<sub>2</sub> were commonly found to increase the corrosivity of the grease [27,34]. However, this study found that the addition of MoS<sub>2</sub> in grease formulation may help in protecting metals from corrosion.

#### 3.8. FTIR characterization of grease

FTIR analysis had been conducted to determine the functional group of grease samples formulated using both ITO and WTO, and with and without the addition of additive, respectively. The measurement was within 500–4000 cm<sup>-1</sup>. Fig. 4 shows the grease samples spectra as the representative of all grease samples. There were no notable differences between all formulated greases.

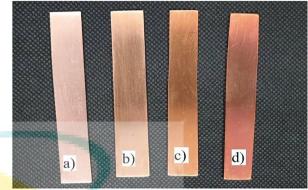
By referring to Fig. 5, all grease samples have high intensity bands in the region of  $2852-2951 \text{ cm}^{-1}$ , as a result of the CH<sub>2</sub> and CH<sub>3</sub> asymmetric stretching. A strong band at  $1455 \text{ cm}^{-1}$  and  $1376 \text{ cm}^{-1}$  represented the C–H asymmetric bending vibrations indicating the functional group of alkane [20,21]. The presence of peaks at  $1098 \text{ cm}^{-1}$  to  $1100 \text{ cm}^{-1}$  were related to the Si-O-Si stretching vibration from the fumed silica thickener [35,36]. A weak band at  $841 \text{ cm}^{-1}$  and  $721 \text{ cm}^{-1}$  represented the C–H out of plane bend indicating the presence of alkanes [22].

Table 5 Corrosivenes	s of base oil.
Sample	Result
ITO WTO	1a – slight tarnish (light orange) 1a – slight tarnish (light orange)

Table 6

 Grease corrosiveness test.

Sample	Result
IG <sub>1</sub>	2a - moderate tarnish (claret red)
$IG_2$	1b – slight tarnish (dark orange)
$WG_1$	1b – slight tarnish (dark orange)
$WG_2$	1b – slight tarnish (dark orange)
WG <sub>3</sub>	1a – slight tarnish (light orange)
$WG_4$	1b – slight tarnish (dark orange)
WG <sub>5</sub>	1a – slight tarnish (light orange)
$WG_6$	1a – slight tarnish (light orange)
WG <sub>7</sub>	1b – slight tarnish (dark orange)
WG <sub>8</sub>	1a – slight tarnish (light orange)
	IG <sub>1</sub> IG <sub>2</sub> WG <sub>1</sub> WG <sub>2</sub> WG <sub>3</sub> WG <sub>4</sub> WG <sub>5</sub> WG <sub>6</sub> WG <sub>7</sub>



**Fig. 4.** Copper strips corrosion a) freshly polished b) class 1a–slightly tarnish c) class 1b–slightly tarnish d) class 2a–moderate tarnish.

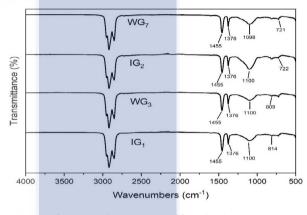


Fig. 5. FTIR characterization of formulated greases.

#### 4. Conclusion

The formulation of grease using WTO is a new idea. Transformer oil is derived from petroleum resources which was in the same categories as lubricating oil. The possibility for WTO being used as new product (grease's base oil) could be a promising base oil alternative to replace the conventional fresh petroleum oil – which currently decreasing due to the declined of petroleum reserves and to create a green product. The physical properties of the formulated FS greases using WTO blended with MoS<sub>2</sub> is encouraging for future application. Further experimental work related to tribology is required to study the performance of the formulated greases for its ability to perform the grease's primary key function.

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#### **Declaration of interest**

None.

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## About Us

MNR Multitech Sdn Bhd was incorporated in 6<sup>th</sup> October 2017 and focused on dealing commercialization of UMP research product, G-Grease.

G-Grease is a multipurpose grease from industrial waste which was developed since 2012.

## Vision

A prominence green technology product based company in Malaysia.

## Mission

Provide world class green technology products, research, analysis, technical services, and consultation towards better environment.



## **Contact Us**

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## **Distributed by**



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## MNR MULTITECH SDN BHD

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# **G-GREASE**





# MNR MULTITECH SDN BHD

**G-Grease** High Performance Grease

## **G-GREASE**

High Performance Grease From Industrial Waste

The product is heavy duty grease from mixture of fresh and recycling which base oil provide high performance at low cost. environmental friendly and safe. Stability, quality and properties of the product have been tested using ASTM (American Society of Testing and Materials) test.

## **ADVANTAGES**

- Readily available contact grease for the maintenance teams
- 2. The contact grease has been proven to be suitable for mechanical equipment.
- 3. High performance at low price as compared to other greases.

Test	Result	Specification
Appearance	Grey	Grey
	Translucent	Ttanslucent
Physical State	Semi-solid	Semi-solid
NLGI Class	2	2-3
Base Oil Type	Mineral	Mineral
Oil Bleeding Test	-9.25	-20 until 40
(% Diff)		
Dropping Point	269.33°C	>170 °C
(°C)		
Contamination	No	No
Test	Contamination	contamination
Penetration Test	260	220-295
@ 25°C (mm)		



Mechanical Grease Worker Double Jacketed Mixing Tank Stability, quality and properties of G-Grease have been tested using ASTM (American Society of Testing and Materials)





Safe

Heavy Duty



## **SPECIFICATION OF GREASE**



Welcome MOHD NAJIB BIN RAZALI

## UIC161004

#### > Research Summary

- > Research Type, FOR & SEO Update
- > View Research Proposal
- > Research Team
- Research Payment Details
- > Research Output
- > Progress & Final Report
- > Download
- > Research Application

Research Project Details				
Project ID	UIC161004			
Project Title	HIGH PERFORMANCE GREASE FROM INDUSTRIAL WASTE			
Project Category	Sains Tulen (Pure Science)			
Project Status	Sedang Berjalan			
Start Date	15/01/2017			
End Date	14/01/2018			
Extension Date	14/07/2019			

Lists of Researcher for UIC161004

No	Staff ID	Staff Name	Start Date	End Date	Position
1	01460	MOHD NAJIB BIN RAZALI	15/01/0017	14/01/0018	Ketua
2	0313	MOHD SABRI BIN MAHMUD	15/01/2017	14/01/2018	Ahli
3	01640	MUSFAFIKRI BIN MUSA	15/01/2017	14/01/2018	Ahli
4	01623	MOHD AIZUDIN BIN ABD AZIZ	15/01/2017	14/01/2018	Ahli
5	0533	ROSLI BIN MOHD YUNUS	15/01/2017	14/01/2018	Ahli

Lists of External Reseacher / Collaborator for UIC161004

**Research Project Financial Info** 

Vote	Approved (RM)	Received (RM)	Expenditure (RM)	Balance (RM)
11000 - SALARY & WAGES	0.00	0.00	0.00	0.00
21000 - TRAVEL & TRANSPORTATION	4,918.17	4,918.17	4,918.17	0.00
22000 - TRANSPORTATION OF GOODS	0.00	0.00	0.00	0.00
23000 - COMMUNICATION & UTILITIES	0.00	0.00	0.00	0.00
24000 - RENTAL	3,000.00	3,000.00	3,000.00	0.00
26000 - SUPPLY OF RAWS MATERIALS	0.00	0.00	0.00	0.00
27000 - RESEARCH MATERIALS & SUPPLIES	20,575.83	20,575.83	20,574.37	1.46
28000 - MAINTENANCE & MINOR REPAIR SERVICES	0.00	0.00	0.00	0.00
29000 - SPECIAL SERVICES (CONFERENCE FEES <= 2500)	25,000.00	25,000.00	25,000.00	0.00
35000 - SPECIAL EQUIPMENT (NOT MORE THA	AN 34,506.00	34,506.00	34,506.00	0.00
Total (RM	A) 88,000.00	88,000.00	87,998.54	1.46

## **RESEARCH OUTPUT**

**Research Exhibition & Award Winning List** 

Publication List

Journal List

**Conference List** 

**Research Intellectual Property List** 

Human Capital Development

Human Capital	Number				
numan Capitai	On-g	oing	Graduated		
Citizen	Malaysian	Non Malaysian	Malaysian	Non Malaysian	
PHD Student	0	0	0	0	
Master Student	0	0	0	0	