

Development of biodegradable lubricating grease for application in specialized equipment operating under marine environmental conditions

Tran Van Hien, Do Thi Thuy*

Institute of Materials, Biology and Environment, Academy of Military Science and Technology, 17 Hoang Sam, Nghia Do, Hanoi, Vietnam.

*Corresponding author: dothuyvlnn@gmail.com

Received 6 Jun. 2025; Revised 29 Jul. 2025; Accepted 10 Aug. 2025; Published 25 Aug. 2025.

DOI: <https://doi.org/10.54939/1859-1043.j.mst.105.2025.67-74>

ABSTRACT

Lubricating greases play a crucial role in protecting and maintaining the stable operation of equipment, particularly under the harsh and corrosive conditions of marine environments. Most currently available greases are formulated from mineral oils, which are non-biodegradable and pose serious environmental pollution risks. In response to the increasing demand for environmentally friendly alternatives, especially in marine environments where various military devices operate, the development of biodegradable lubricating greases has become a necessity. This study presents the synthesis and evaluation of a biodegradable grease formulated using castor oil as the base oil, lithium 12-hydroxystearate soap as the thickener, and functional additives with anti-corrosion and anti-wear properties. The grease was prepared with a thickener-to-base oil ratio of 14:86 wt.% and temperature processed at 220–230 °C, resulting in a stable structure and high homogeneity. Salt spray test results demonstrated a significant improvement in metal surface protection. Additionally, the grease exhibited a load-carrying capacity of 315 kg and a biodegradability exceeding 70%. The results indicate that the grease offers excellent lubricating and protective properties and high biodegradability, making it suitable for equipment operating in marine environments.

Keywords: Lubricating grease; Biodegradation; Friction; Wear.

1. INTRODUCTION

Lubricating grease is a colloidal system composed of three primary components: base oil, thickener, and performance additives. Typically existing in a semi-liquid to semi-solid state, grease is widely applied in mechanical systems to reduce friction, minimize wear, protect metal surfaces, and prevent the entry of moisture and contaminants. The characteristic properties of grease arise from the synergistic interaction among its three components. Specifically, the thickener determines the consistency and structural framework of the grease. At the same time, additives enhance properties such as oxidation resistance, thermal stability, anti-rust performance, corrosion inhibition, and high-pressure resistance [1].

The base oil is typically a petroleum-derived mineral in conventional lubricating greases due to its favorable stability, high lubricating efficiency, and low cost. However, mineral oils exhibit very low biodegradability, with reported degradation rates typically ranging from 15% to 35% under standard test conditions [2]. When released into the environment, particularly marine ecosystems, they can cause persistent pollution and severely impact aquatic life [3]. Studies have also demonstrated that residual hydrocarbons from non-biodegraded lubricants can induce chronic toxicity in fish, crustaceans, and planktonic organisms, while also bioaccumulating through food chains and posing long-term risks to human health.

Considering growing environmental threats and the global push for sustainable development, there has been a marked change in the lubricant industry toward using both renewable and readily biodegradable base oils. Vegetable oils, which contain unsaturated fatty acid chains in their molecular structure, exhibit excellent lubricity, appropriate viscosity, and chemical compatibility

with a variety of thickeners such as lithium compounds, polyurea, modified bentonite, and saboxite [4]. Moreover, bio-based oils offer economic advantages by utilizing agricultural byproducts, reducing dependence on fossil resources, and promoting circular economy models. Several recent studies have demonstrated the potential of high-performance biodegradable greases. Campanella et al. synthesized a biodegradable grease using epoxidized canola oil and lithium hydroxystearate as the thickener, showing favorable mechanical performance and thermal stability in the temperature range of - 20 to 120 °C [5]. Padgurskas et al. investigated the formulation of a biodegradable lubricating grease based on rapeseed oil, employing beeswax as the thickening agent. The grease was further modified with functional anti-wear additives, antioxidants, and carbon-based nanoparticles [6]. Sapnar et al. developed a biodegradable lubricating grease using palm oil as the base oil. The formulation combined palm oil with stearic acid, calcium hydroxide, glycerol monostearate, soy wax, and hydroxyapatite to produce a high-performance lubricant with significant ecological advantages. In the formulation, the selected components acted as saponifying agents, thickeners, and functional additives, contributing to improvements in consistency, structural stability, and tribological behavior [7]. In Vietnam, research and application of biodegradable lubricating greases have received increasing attention in recent years [8, 9].

This study reports the formulation of a biodegradable lubricating grease using castor oil, a selected thickener, and functional additives. Preliminary evaluations assessed its corrosion protection, anti-wear performance, and biodegradation potential under laboratory conditions. Initial findings suggest that the developed grease holds promise for application in military equipment operating under harsh marine environments.

2. EXPERIMENT

2.1. Materials

- Base oil: Castor oil (India) was employed as the base fluid. Its key physicochemical properties included a specific gravity of 0.96 g/mL, saponification value of 182 mg KOH/g, kinematic viscosity of 250 cSt at 40 °C, and flash point of 290 °C.

- Thickener: Lithium 12-hydroxystearate was utilized as the thickening agent. This compound was synthesized in-house via a conventional saponification process using 12-hydroxystearic acid (analytical grade, Macklin, China) as the starting material.

- Additives: To enhance the functional performance of the grease, the following additives-supplied as pure chemicals by Macklin (China)-were incorporated: diphenylamine P89 as an antioxidant and anti-rust agent; zinc dialkyldithiophosphate (ZnDTP) as an anti-wear additive; and a combination of molybdenum disulfide (MoS₂) and graphite as solid lubricants to reduce friction.

- Steel (CT3), aluminum (Al), and copper (Cu) plates with dimensions of 10 × 10 cm were selected as the materials for corrosion investigation in this study.

- Steel wire specimens with lengths of 0.5 – 1.0 m were selected for field testing under realistic operational conditions.

2.2. Experimental

Biodegradable lubricating grease was prepared in four sequential stages. In the first stage, lithium 12-hydroxystearate soap was synthesized in-house by reacting 12-hydroxystearic acid with lithium hydroxide at 90 - 95 °C under continuous stirring. In the second stage, the synthesized soap was dispersed into castor oil at an elevated temperature of 200 - 210 °C to form a homogeneous mixture. In the third stage, the resulting mixture was gradually cooled to 120 °C to allow grease crystallization and structural development. In the fourth stage, functional additives-including diphenylamine P89, ZnDTP, MoS₂, and graphite-were incorporated at 120 - 130 °C under constant stirring. Each additive was added at a concentration of 0.05 wt.%, resulting in a total additive

content of 2 wt.% in the final grease formulation. Following additive incorporation, the grease was further stirred to ensure uniform distribution and homogenized using a colloid mill to obtain a consistent and stable final product. Five grease samples (M1, M2, M3, M4, M5) were prepared with lithium 12-hydroxystearate contents of 10, 12, 14, 16, and 18 wt.% in castor oil, respectively. Their dropping point and working penetration were evaluated using standard methods.

2.3. Materials characterization

- Corrosion resistance was evaluated through accelerated salt spray testing by ASTM B117. Test specimens included carbon steel (CT3, Fe), copper (Cu), and aluminum (Al) plates. Before testing, all metal surfaces were polished using emery paper to achieve a surface finish equivalent to triangular gloss grade 8, followed by uniform application of the prepared grease. A blank sample (without additives) was also included as a control for comparative analysis.

- Anti-wear performance was assessed using the four-ball wear test method by ASTM D2596-02. The test was conducted under standard conditions to determine the wear-preventive characteristics of the formulated greases.

- The biodegradability of the lubricating grease was evaluated using the OECD 306 method. The primary objective was to measure the dissolved organic carbon (DOC) concentration in the solution after 28 days of testing. Biodegradation was assessed by monitoring the reduction in DOC compared to the initial concentration, thereby determining the percentage of organic matter degraded. All tests were conducted under controlled conditions, with the temperature maintained between 15 ± 2 °C and 20 ± 2 °C, mild stirring, and each test repeated at least three times to confirm that results are reliable and repeatable.

2.4. Field testing under marine conditions

Assessment of corrosion protection under marine climatic conditions

The corrosion resistance was evaluated through direct exposure tests conducted in Cam Ranh, Khanh Hoa, Vietnam. Test specimens comprised steel wire ropes with 10 mm, 20 mm, and 32 mm diameters. Each rope sample was uniformly coated with a one mm-thick layer of the formulated grease. The specimens were suspended in seawater at the port area and fully submerged for 60 days to simulate long-term marine exposure. A schematic diagram of the experimental procedure is presented in figure 1.

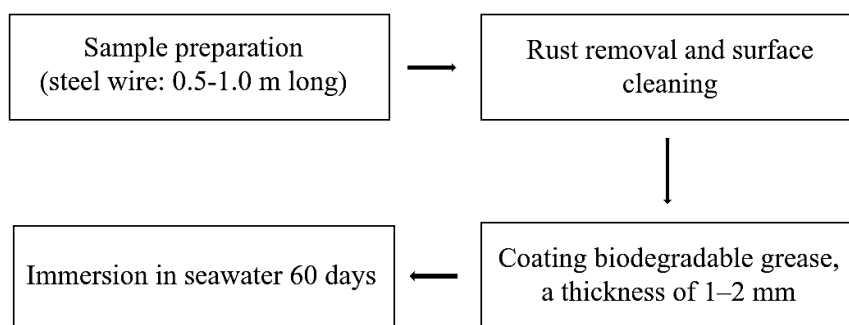


Figure 1. Schematic diagram of the experimental procedure for evaluating the corrosion resistance of biodegradable grease coating.

Performance testing on the 4500-ton lifting platform

Performance testing was carried out on a 4500-ton lifting platform in a salt-laden, high-humidity marine environment. Steel wire ropes coated with biodegradable grease underwent alternating seawater immersion and chloride-rich air exposure during operation. The ropes faced dynamic loads and environmental fluctuations. After testing, the grease was assessed for adhesion, load-bearing capacity, and friction reduction. A schematic is shown in figure 2.

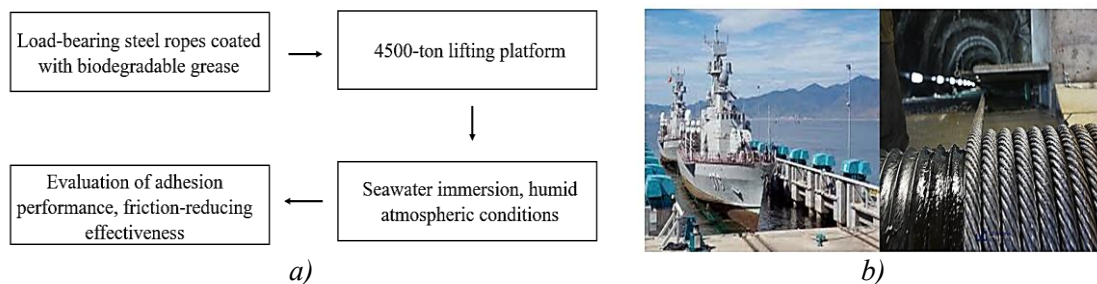


Figure 2. (a) Evaluation process and (b) field images of biodegradable grease on steel ropes in a 4500-ton lifting platform.

3. RESULTS AND DISCUSSION

3.1. Effect of dispersed phase and dispersion medium

To investigate the influence of the dispersed phase and the dispersion medium on the structural formation and stability of biodegradable lubricating grease, a series of formulations was prepared by varying the mass ratio between lithium 12-hydroxystearate (dispersed phase) and castor oil (dispersion medium). The physicochemical properties of the prepared greases as a function of the dispersed phase concentration, the results are presented in figure 3.

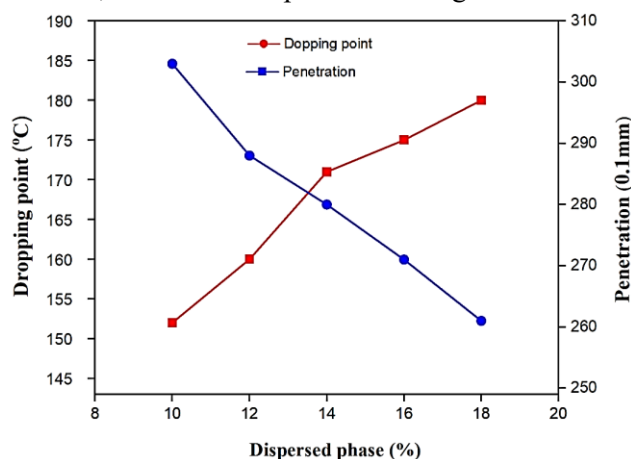


Figure 3. Effect of lithium 12-hydroxystearate concentration on the properties of greases.

The results presented in figure 3 indicate that the dispersed phase content significantly affects the physical properties of the lubricating grease, particularly concerning penetration and dropping point. Samples M1 and M2, formulated with relatively low concentrations of lithium 12-hydroxystearate (10 - 12 wt.%), exhibited high penetration values and low dropping points, suggesting a soft consistency and limited thermal resistance. These characteristics are generally undesirable in high-performance lubricants, as they reflect poor structural strength and low temperature resistance. With increasing thickener content, samples M3 and M4 showed a marked improvement in rheological behavior. Specifically, the penetration values decreased while the dropping points increased, indicating enhanced structural integrity and thermal stability. Sample M3 exhibited a homogeneous brownish-yellow appearance, good consistency, and no observable phase separation or oil release, suggesting excellent formulation homogeneity. Although sample M5 achieved the highest dropping point (180 °C), it also demonstrated a significant reduction in penetration, indicative of excessive hardness. This may be caused by the thickener content being higher than the optimal level, which reduces the uniformity of the grease structure and limits the dispersion ability of the system. A low thickener concentration produces a thermally unstable, overly soft product, while an excessive

amount produces a substantial, less workable material. Based on the comprehensive evaluation, the formulation corresponding to sample M3 (14 wt.% lithium soap/86 wt.% castor oil) was identified as a suitable composition for further development and application.

3.2. Effect of processing temperature

The development of the grease microstructure is strongly influenced by thermal treatment during synthesis, wherein lithium 12-hydroxystearate is dispersed in castor oil at high temperatures. To identify the optimal temperature range for the heat treatment stage, experiments were conducted on grease sample M3, with processing temperatures ranging from 190 °C to 250 °C. The evaluated parameters included the dropping point and penetration. The results are shown in figure 4.

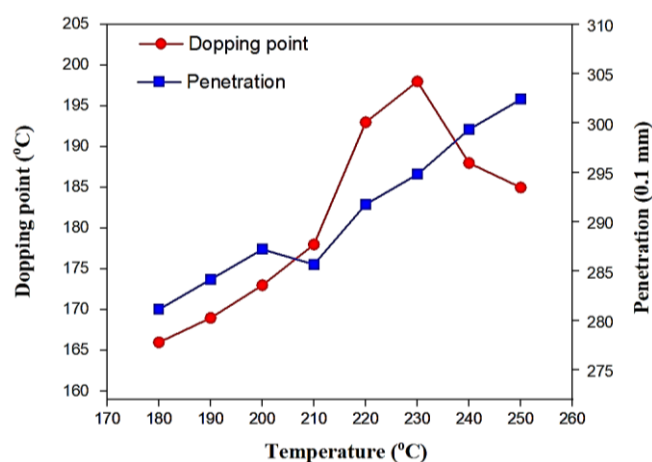


Figure 4. Effect of synthesis temperature on the physical properties of the grease.

As shown in figure 4, grease samples processed at 220 - 230 °C exhibited the highest dropping points (193 - 198 °C) and maintained relatively stable penetration values (288 - 290 × 0.1 mm). These results indicate the formation of a dense, well-developed, and thermally stable soap fiber network. In contrast, samples synthesized at lower temperatures (180 - 190 °C) showed insufficient soap fiber formation, as reflected by lower dropping points and penetration values, suggesting a softer structure and reduced thermal stability. Conversely, samples processed at higher temperatures (240 - 250 °C) exhibited a notable decrease in dropping point and increased penetration values. This trend may be attributed to partial degradation of the soap network under excessive thermal conditions, which adversely affects the structural integrity of the grease. Based on these findings, the temperature range of 220 - 230 °C was selected for grease synthesis in subsequent experimental investigations.

3.3. Performance evaluation of the biodegradable lubricating grease

Corrosion protection performance under salt spray accelerated testing

The corrosion resistance of the biodegradable lubricating grease was evaluated through an accelerated salt spray test conducted by ASTM B117. The test includes 20 spray cycles, and the corrosion levels were assessed using the Rust Evaluation (Re) index, which ranges from Re0 (no visible rust) to Re10 (severe corrosion, > 90% surface). The results are presented in table 1.

As shown in table 1, the uncoated metal specimens (blank samples) exhibited severe corrosion (Re10) after only five test cycles for all three metals (CT3 steel, Cu, and Al), indicating extensive surface degradation (90–97% rust coverage) and poor natural corrosion resistance under rich salt conditions. By contrast, metal samples coated with the formulated biodegradable grease showed significantly improved corrosion resistance throughout the 20-cycle test. In the case of CT3 steel, the rust severity progressed from Re2 (approximately 2–4% surface corrosion) at cycle 5 to Re7

(approximately 30–35%) at cycle 20. Copper samples exhibited Re3 to Re8 during the same interval, while Aluminum samples showed a more stable behavior, maintaining rust grades between Re3 and Re7. The improved performance can be attributed to corrosion-inhibiting additives in the grease formulation, which likely form a protective barrier at the metal–grease interface and suppress electrochemical degradation processes.

Table 1. Corrosion performance of grease samples under salt spray conditions.

Material	Assessment after each cycle			
	5	10	15	20
CT3 steel (uncoated)	Re10	-	-	-
CT3 steel-coated grease film	Re2	Re3	Re5	Re7
Cu (uncoated)	Re10	-	-	-
Cu-coated grease film	Re3	Re4	Re6	Re 8
Al (uncoated)	Re10	-	-	-
Al-coated grease film	Re3	Re4	Re5	Re7

Anti-wear performance evaluation

The anti-wear performance of the biodegradable lubricating grease was assessed using the four-ball test method by ASTM D2596-02. The base grease formulation contained no functional additives and exhibited a weld load of 192 kg. In contrast, the grease sample formulated with anti-wear additives demonstrated a significantly improved weld load of 315 kg. This corresponds to a 64.06% enhancement in load-carrying capacity, indicating the substantial effect of the additives on reinforcing the mechanical strength of the lubricating film formed between contacting surfaces. The observed improvement is primarily attributed to the selective adsorption of additive molecules onto the metal substrate, which facilitates the formation of a protective boundary layer. This layer effectively minimizes direct metal-to-metal contact, reduces local mechanical interaction, and restricts plastic deformation under high stress conditions. These results highlight the superior lubricating performance of the grease system based on vegetable oil and thickened with lithium 12-hydroxystearate, when combined with appropriate anti-wear additives.

Biodegradability assessment

The grease biodegradability was assessed using OECD 306, based on dissolved organic carbon reduction in simulated marine conditions. Results for the four samples are shown in table 2.

Table 2. Biodegradability of lubricating grease samples.

Sample	Initial DOC (mg/L)	Final DOC (mg/L)	Biodegradation degree (%)
M1	120.0	34.5	71.3
M2	115.0	34.3	70.2
M3	118.0	32.9	72.1
M4	122.5	33.7	72.5

The results presented in table 2 indicate that the grease sample prepared with castor oil as the dispersion medium achieved a biodegradation rate exceeding 70%. According to OECD classification standards, this level qualifies as readily biodegradable, indicating high environmental friendliness. These results affirm the suitability of castor oil-based grease formulations for applications where biodegradability and ecological safety are critical, particularly in marine or outdoor environments where lubricant loss may result in direct environmental exposure.

Field testing under marine conditions

In the first testing, grease-coated steel cables were immersed at 2 meters in natural seawater for 60 days. The grease film retained excellent adhesion across the cable surface throughout the testing period without any signs of flow, delamination, or coating loss. No discoloration or degradation was observed, indicating high chemical stability in chloride-rich environments. Additionally, the absence of visible rust or corrosion on the underlying metal surface confirmed the effectiveness of the grease in inhibiting corrosion. These results show that the biodegradable grease retains morphological stability, adhesion, and protective performance under prolonged exposure to harsh marine conditions, underscoring its suitability for maritime and coastal defense applications.

In the second testing, the grease was applied to load-bearing cables of a 4500-ton ship-lifting platform. The system operated continuously over three months under combined static and dynamic testing in a marine environment. The grease exhibited high lubricity, significantly reducing friction and wear, while resisting mechanical stress without oil separation or loss of adhesion. It maintained morphological stability and surface coverage throughout the operating cycle, even under high humidity, saline exposure, and mechanical loading. These findings further confirm the durability and multifunctional performance of the grease, supporting its applicability in harsh operational environments such as naval infrastructure and heavy-duty maritime systems.

5. CONCLUSIONS

This study successfully formulated a biodegradable lubricating grease tailored for extreme marine environments. Field testing on a 4500-ton ship-lifting platform confirmed its strong adhesion, stable surface coverage, and effective friction and wear reduction under combined static and dynamic loading in seawater. In addition to its mechanical and tribological performance, the grease offers significant environmental advantages due to its biodegradability, minimizing ecological risks associated with conventional lubricants. Overall, the results confirm its practical applicability in naval and heavy-duty maritime systems and emphasize its potential as a sustainable alternative to traditional mineral oil-based greases. Further investigations into its lifecycle performance and compatibility with various naval components will support broader deployment in sustainable maintenance practices for maritime defense systems.

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TÓM TẮT

Nghiên cứu chế tạo mỡ bôi trơn có khả năng phân hủy sinh học ứng dụng cho thiết bị chuyên dụng hoạt động trong môi trường biển

Mỡ bôi trơn đóng vai trò quan trọng trong việc bảo vệ và duy trì hoạt động ổn định của các thiết bị, đặc biệt là trong điều kiện môi trường biển khắc nghiệt. Tuy nhiên, phần lớn các loại mỡ hiện nay được sản xuất từ dầu khoáng không có khả năng phân hủy sinh học, tiềm ẩn nguy cơ ô nhiễm môi trường nghiêm trọng. Trước nhu cầu phát triển các sản phẩm thân thiện với môi trường, đặc biệt trong điều kiện các thiết bị chuyên dụng thường xuyên vận hành tại khu vực biển đảo, việc nghiên cứu chế tạo mỡ bôi trơn phân hủy sinh học là hết sức cần thiết. Bài báo này trình bày kết quả nghiên cứu tổng hợp và đánh giá mỡ bôi trơn có khả năng phân hủy sinh học sử dụng dầu thầu dầu là dầu nền, chất làm đặc là xà phòng lithium 12-hydroxystearat, kết hợp với hệ phụ gia chức năng chống gỉ, chống mài mòn. Mỡ được chế tạo với tỷ lệ chất làm đặc/dầu nền là 14/86% khối lượng và nhiệt độ xử lý 220-230 °C cho cấu trúc bền và độ đồng đều cao. Kết quả thử nghiệm mù muối chứng minh mỡ phủ giúp cải thiện rõ rệt khả năng bảo vệ bề mặt kim loại. Ngoài ra, khả năng chịu tải của mỡ đạt 315 kg và khả năng phân hủy sinh học vượt 70%. Những kết quả này cho thấy mỡ có khả năng bôi trơn, bảo vệ kim loại và có khả năng phân hủy sinh học cao, đáp ứng yêu cầu kỹ thuật đối với các thiết bị hoạt động trong môi trường biển.

Từ khoá: Mỡ bôi trơn; Phân hủy sinh học; Ma sát; Mài mòn.