



# Removal of greases and lubricating oils from metal parts of machinery processes by subcritical water treatment

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## ARTICLE INFO

### Article history:

Received 27 January 2013

Received in revised form 26 March 2013

Accepted 28 March 2013

### Keywords:

Degreasing

Grease

Lubricant

Machinery parts

Subcritical water

Environmentally benign solvent

## ABSTRACT

Toxic or persistent solvents have been widely used to remove greases and lubricants from various machine elements in the washing processes. In this study, an alternative degreasing method that employed subcritical state water was assessed. This environmentally benign solvent has significant potential for various degreasing applications. The operation time and temperature and flow rate of subcritical water had markedly positive impacts on the degreasing efficiency. However, the effect of pressure of subcritical water flow was minimal. The degreasing efficiency was also highly dependent on the physical characteristics and chemical composition of grease. The subcritical water treatment demonstrated a better degreasing efficiency than conventional degreasing methods. Only minor physical damage was observed on the metal parts after the subcritical water treatment. Conclusively, it was found that the subcritical water degreasing system can be used as an effective degreasing technology for machinery operations.

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## 1. Introduction

Greases and lubricating oils are ubiquitously used in machine-related processes and operations, providing a fluid layer to separate moving surfaces of machines, minimizing heat and friction, and hindering surface wear under extreme temperature and pressure conditions [1]. These materials are typically mixtures of alkanes, alkenes, alicyclics, and aromatics distilled from crude oil and then modified with various thickeners and other additives to impart the characteristics required for specific applications [2,3]. Proper removal of these products from the metal parts after use is necessary for the continuous and repetitive operation of machinery processes [4]. It is, in fact, not easy to remove greases and lubricating oils from metal surfaces during production or machinery repair since they are complex mixtures designed to resist degradation during strenuous use [5].

Typical multipurpose greases contain about 85% base oil, 10% thickener, and 5% additives [1]. The most common base oils are mineral oils, but synthetic oils are also popular for special grease applications. The chemical composition of mineral oils is not easily defined because they can contain various hydrocarbons depending on the sources of crude oils specific to origin and extent of refining. Synthetic hydrocarbons such as polyalphaolefins, alkylated

aromatics, polyglycols, ester and silicone oils, and perfluoropolyethers, are more clearly defined with regard to their molecular weight and structure. The second most abundant component of grease is the thickener. Thickeners are finely dispersed in the base oils, forming a structure similar to an emulsion and these substances primarily determine the physical property of the grease [2]. Soaps of lithium, calcium, sodium, or aluminum are the most common thickeners, but solid particles such as bentonite clay, silica, graphite, and polymers are also employed. The third main components of grease are additives, including anti-oxidants, anti-wear additives, and friction and structure modifiers.

Conventionally, combinations of cleaning and rinsing baths, mechanical treatment (ultrasound, spraying, and injection flood washing), and drying (hot air, vacuum, and denaturing fluids) have been used, depending on specific applications and the nature of the items that require decoating [3,5]. The most common solvent media used for degreasing can be classified as follows: organic solvents, strong alkalis (e.g., sodium hydroxide), alkali salts (e.g., cyanides and borates), complexing agents (e.g., EDTA and NTA), surfactants (e.g., nonylphenyl), and corrosion inhibitors (e.g., sodium benzoate). Among them, organic solvents are known to be hazardous to both human health and the environment, and in addition, some of them are potential or known carcinogens [5]: e.g., hexane, trichloroethane, trichloroethylene (TCE), tetrachloroethylene, chloro-fluoro-carbon 113, and dichloromethane (DCM). The aqueous-based solvents are usually strongly alkaline or acidic in nature and therefore highly toxic and corrosive [3].

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Subcritical water represented by superheated liquid (water heated to any temperature less than its critical temperature of 373 °C with enough pressure to maintain its liquid state) has been explored in a number of prior studies [6,7]. This environmentally benign solvent has significant potential for beneficial use in a wide variety of degreasing and decoating applications [8]. Subcritical water can dissolve polar and non-polar organic compounds that are insoluble under ambient conditions because its dielectric constant ( $\epsilon$ ) decreases from ~80 at room temperature to ~30 at 250 °C [9,10]. At 300 °C, the density and  $\epsilon$  of water approach those of ambient acetone [10]. As a result, the solubilities of hydrophobic organic compounds in the subcritical water can be increased significantly by raising the temperature [11,12]. Another benefit of subcritical water for degreasing is the potential for recovery, recycling, and reprocessing of valuable organic chemicals, organic solvents, and greases [13].

In this study, the development of an alternative degreasing method that employs essentially pure water was explored. Specifically, the removal of grease and lubricating oil products from metal parts using a subcritical water extraction system was evaluated. Tests were performed on a variety of greases and lubricating oils under varying operating conditions. The degreasing efficiency of the subcritical water treatment was compared with those obtained by using conventional degreasing and cleaning agents. We also investigated the corrosion potential of subcritical water for future use. The results of this study are expected to demonstrate the general applicability of the subcritical water degreasing system.

## 2. Materials and methods

### 2.1. Materials

Greases and lubricating oils, chosen based on their application and components, were purchased from local mechanic shops (Table 1). Organic solvents (hexane, DCM, TCE, and methanol) and Zep cleaner used as a conventional degreasing agent were purchased from Fisher Scientific (Chicago, IL, USA) and Zep Superior Solutions (Cartersville, GA, USA), respectively. Petroleum hydrocarbon standard and alkane hydrocarbons (pentadecane [C<sub>15</sub>H<sub>32</sub>], eicosane [C<sub>20</sub>H<sub>42</sub>], and triacontane [C<sub>30</sub>H<sub>62</sub>]) used as internal

standards for gas chromatography (GC) analysis were obtained from Sigma–Aldrich (Milwaukee, WI, USA).

### 2.2. Subcritical water treatment system setup

The degreasing experiments were carried out with the continuous flow subcritical water extraction system shown in Fig. 1. Deionized and deoxygenated (pre-purged with helium) water was pumped through a preheat coil (stainless steel tubing of 1.6-mm o.d., 0.5-mm i.d., and 3.5-m length) located in a temperature programmable GC oven (HP Model 5890, Hewlett Packard, Houston, TX, USA) using a high performance liquid chromatography (HPLC) pump (Constant-Flow HPLC Pump, Cole-Parmer, Vernon Hills, IL, USA). The subcritical water (or steam) was then passed through a stainless steel extraction cell (9.4-mm i.d. and 90-mm length, 6.315-mL total volume, Alltech, Deerfield, IL, USA). The cell was almost fully filled with metal ball bearings (4.75-mm o.d.), and an aliquot (~0.03 g) of grease or lubricating oil was uniformly applied on their surfaces. After the subcritical water (or steam) left from the oven, it passed through a cooling coil to drop the effluent temperature to the ambient temperature. For subcritical water operation, the system was pressurized by controlling the tubing line with a back-pressure regulator (Tescom, Elk River, MN, USA). After the extraction, the ball bearings were collected from the extraction cell. Effects of run time (1–90 min), temperature (100–250 °C), pressure (10.3–37.9 bar), and flow rate (0.5–5 mL/min) on the degreasing efficiency were assessed to find the best-optimized operating condition. For steam operation, the system was not pressurized and the back-pressure regulator was left open.

### 2.3. Degreasing with conventional solvents

Conventional degreasing agents (hexane, DCM, TCE, methanol, and Zep cleaner) were used to compare their degreasing efficiencies with those obtained by the subcritical water and steam degreasing systems. The reactor setup for this experiment was the same as that for the subcritical water treatment system. Each degreasing agent fluid was passed through the extraction cell at 2 mL/min for 60 min with neither heating nor pressurizing. After the extraction, the ball bearings were collected from the extraction cell and the degreasing efficiency was quantified.

**Table 1**  
Greases and lubricating oils and their defining characteristics.

Grease/oil name	Base oil type	Thickener/additive	Dropping point (°C)	Viscosity at 100 °C cSt (ASTM D 445)	Applications
Army Grease	Hydrocarbon	Lithium complex	260+	N/A	Military lubricating grease
AeroShell #5	High viscosity mineral	Microgel® (clay)	260+	31.8	High temp. aircraft wheel bearing and engine accessory grease
AeroShell #6	Mineral	Microgel® (clay)	260+	5.5	General purpose airframe grease used in plain and anti-friction bearings
AeroShell #14	Mineral	Calcium	150	3.1	Helicopter grease, main and tail rotor bearings
AeroShell #17	Synthetic diester	Microgel® (clay)	260+	3.1	Extreme pressure multipurpose grease for heavily loaded sliding steel surfaces, bogie pivot pins on jet aircraft landing gear assemblies
AeroShell #22	Synthetic hydrocarbon	Microgel® (clay)	260+	5.8	Aircraft wheel bearings, engine accessories and airframe lubrication
Castrol 10W-30	Hydrocarbon	None	N/A	N/A	Motor engine oil (petroleum base oil)
Castrol 10W-30 [Syntec Blend]	Synthetic hydrocarbon	None	N/A	N/A	Motor oil (synthetic base oil)
Texhvi 3	Light paraffinic mineral Oil	None	204	3.31	Base Oil
Texhvi 4	Heavy paraffinic mineral Oil	None	210	4.0	Base Oil
Chrysler ATF	Mix Texhvi3/4	N/A	N/A	N/A	Automatic transmission oil (synthetic base oil)

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