

# Glycerol-based hydraulic fluid for hydropower industry

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## Abstract

The project has been running well during the whole project period. All project partners are quite active in the project. The project is run and follows the work plan very well. During the project period, we have done work on lab studies to improve the hydraulic fluid performance, field test, hydraulic fluid quality control methods development, and waste hydraulic fluid treatment methods development. The field tests were not only done at previously planned partners, i.e., Statkraft and Vattenfall. We also managed to convince Uniper to test our hydraulic fluid. All tests went very well, and we gained good knowledge through these field tests. In general, it can be said that glycerol-based hydraulic fluid fits well with the current hydraulic systems. Until now, the results indicate that glycerol-based hydraulic fluid can replace mineral oil-based hydraulics, resulting in less energy consumption and lower hydraulic fluid temperature in the system. Above all, after changing to our glycerol-based hydraulic fluid, the best benefit for the hydropower industry is that they do not need to worry about the hydraulic fluid oils polluting the environment, especially the rivers.

## Publication list

### (1) Peer-reviewed articles in scientific journals

- Paul Okhiria, Marcus Björling, Pontus Johansson, Mushfiq Hasan, Roland Larsson, **Yijun Shi**. Tribological Performance of Glycerol-Based Hydraulic Fluid Under Low-Temperature Conditions. ***Lubricants***, 12 (12): 430 (2024).
- Juan Guillermo Zapata Tamayo, Marcus Björling, **Yijun Shi**, Jens Hardell, Roland Larsson. Micropitting performance and friction behaviour of DLC coated bearing steel surfaces: On the influence of Glycerol-based lubricants. ***Tribology International***, 196, 109674 (2024).

### (2) Articles in popular science journals

- Growing interest in glycerol in hydropower, Energiforsk, 2024-03-04, <https://svc.energiforsk.se/en/news/growing-interest-in-glycerol-in-hydropower/>

### (3) Scientific conference contributions

- **Yijun Shi**. China-Sweden Tribology Symposium, 22<sup>th</sup> November, Lanzhou, P.R. China (Oral presentation) (2023).
- Pontus Johansson, Marcus Björling, Pär Marklund, **Yijun Shi**. NORDTRIB 2022, Ålesund, Norway June 14<sup>th</sup>-17<sup>th</sup> (Oral presentation) (2022).

#### (4) Conference contributions

- Jun Zhao, **Yijun Shi**. Tribology days 2023, 15<sup>th</sup> – 16<sup>th</sup> November, CARL BECHEM GMBH in Germany (Oral presentation) (2023).
- Zhipeng Wu, **Yijun Shi**. Tribology days 2023, 15<sup>th</sup> – 16<sup>th</sup> November, CARL BECHEM GMBH in Germany (Oral presentation) (2023).

### **Deliveries and milestones**

(1) 1 Doctor, Di Wang, defended in September 2024.

(2) Reference group meetings: 3

- a. 2022-11-02: representatives from Sveaskog, Piteå Energi, Vattenfall
- b. 2023-10-11: representatives from Sveaskog, Piteå Energi, Vattenfall
- c. 2024-12-06: representatives from Piteå Energi, Vattenfall, Statkraft

(3) Courses: 2

- a. Spring in 2023: one lecture within the course of Tribology in LTU
- b. Spring in 2024: one lecture within the course of Tribology in LTU

(4) Seminars: 3

- a. 2023-11-07: discussion with Fortum
- b. 2023-11-21: discussion with Skellefteå Kraft
- c. 2024-02-21: discussion with Andritz Hydro GmbH

### **Custom deliverables and milestones**

(1) Cleaning strategy: 1

(2) Service life: 1

(3) Quality monitoring: 1

(4) Waste treatment method: 1

Note: A detailed description of “Custom deliverables and milestones” can be found in the following section of “Experiment content”.

### **Experiment content**

#### **1. Field testing**

##### *Case 1: field test with Svea Skog*

Under the collaboration between Skogsforsk, Plantskolan, Sveaskog, Sustainalube and LTU, the developed hydraulic fluid has been tested in Plantskolan Vibytorps Wheel loader which model is a Lundberg 5200 from the autumn 2022.

**The equipment and material used are listed below:**

- 1x 200 Barrel Cleaning fluid ( Approximately 60-70% were used)
- 1x 200 Litre Barrel Hydraulic fluid (Approximately 60-70% were used)
- 8 Test bottles
- Pneumatic driven pump
- Waste fluid container.

### **Flushing**

The flushing procedure were conducted in the order list below:

- (1) One sample each of clean Flushing fluid and clean Hydraulic fluid were taken in a plastic bottle.
- (2) The tank on the wheel loader were filled with cleaning fluid, the first time it took more fluid since the system were empty.



Fig 1. Leif is filling the first batch of flushing fluid in the hydraulic tank.

- (3) An air pump was used on the tank to fill the pumps (since it had been emptied)



Fig 2. The air pump was pressed onto the tank and hold when running to make sure the pumps were not running dry during the first start-up.

Since the batteries on the wheel loader were completely drained the diesel engine would not start, even with 2 battery chargers connected. But with new batteries the engine started right away. This took around 3 hours since the batteries had to be bought and picked up in Örebro, 20 km from the workshop.



- (4) The wheel loader were started and all hydraulic functions was operated for maybe 3 minutes
- (5) The system was emptied.
- (6) One sample of used Flushing fluid were taken in a bottle
- (7) Fill the system again 60-70% with flushing fluid and repeat step 4-6.

This step (7) was done 2 times. So, in total, the system was flushed 3 times with the cleaning fluid. The forward and backward drive did not work with the cleaning fluid, probably because of its low viscosity.

- (8) The tank was filled with hydraulic fluid to around 60-70% of the tank volume.
- (9) The wheel loader was started, and all hydraulic functions was operated for maybe 10 minutes when driving around on the yard. With the hydraulic fluid in the system the forward and backward drive of the machine worked fine.

- (10) Take one sample of the used “flushing hydraulic fluid”.

- (11) Step 8-10 were repeated with the hydraulic fluid to flush out all Cleaning fluid.

This were done 2 times to make sure all contaminants and cleaning fluid were out of the system.

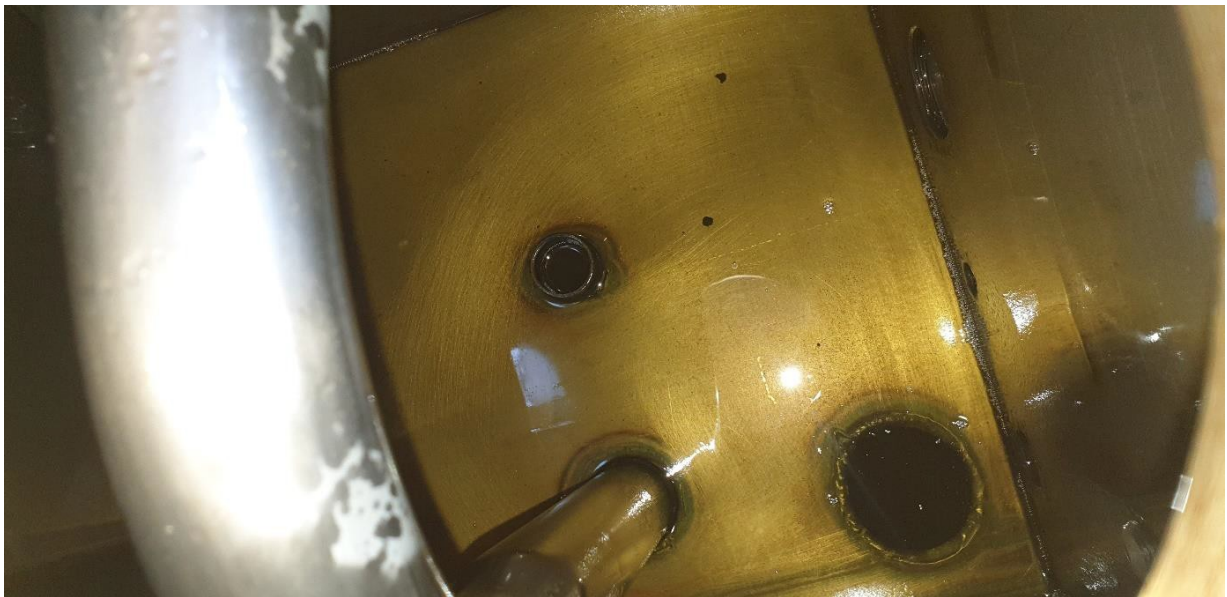


Fig 3. The hydraulic fluid in the tank, after the first flush with hydraulic fluid.

- (12) Now fill the system properly with the hydraulic fluid.
- (13) Later on, the mechanic said he was going to change the oil filter the next day.
- (14) Two stickers were placed on the fluid tank so that no one should fill it with another fluid by mistake (Fig. 4).

Here the notion is wrong as Sustainalubes hydraulic fluid should not be considered as an oil, but the mechanic was determined that otherwise the operators might not understand the message.



Fig 4. Hydraulic fluid tank with warning decals on, also wrong notion here with “oil” but the message should be clear: Only use Sustainalube

The samples of hydraulic fluid (1) after filling into the machine; (2) after around 4 months running with around 550 hours real running hours, were taken, and will be analyzed. Due to the damaged sealing material (Figure 5 and Figure 6), the test is stopped after around 18 months trying.

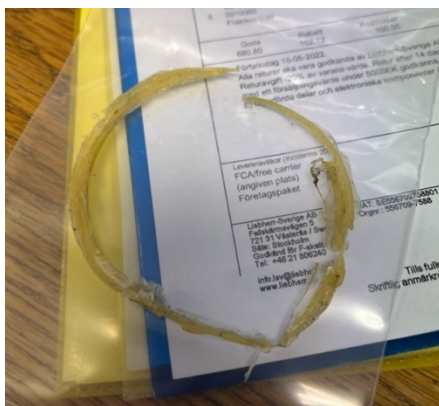


Figure 1, Broken gasket for the hydrostatic pump.



Figure 2, Broken seal on brake cylinder.

After that, we send the pump and motor to the manufacturer (Danfoss) at Denmark for a deep

analysis. The full report from Danfoss is attached. In summary: None of the parts showed any signs of immediate failure, but all the moving parts (piston ball slipper or the valve) showed some sticky behavior and were difficult to move. The valve segment cylinder block interface of the motor exhibits evidence of high-temperature generation. However, in the raceway predominantly polishing wear was observed only. The endcap valve segment interface exhibits adhesive wear, which has a negative influence on the performance of the motor. It is difficult to judge whether this was caused by the long period of storage and subsequent cleaning of the parts, or whether it occurred during operation.

### *Case 2: field test in Vattenfall*

Under the financial support by previous Håva project funded by Energimyndigheten (Håva, Dnr 2018-003910), a field test has been started in Vattenfall R&D center in Älvkarleby since 2021.07 until now. Since Vattenfall is so happy with the performance, Vattenfall changed another hydraulic system which they bought in the summer of 2022 into our glycerol-based hydraulic in 2022-09-29. Two hydraulic systems are keep running under a temperature of around 50 to 60 °C and working pressure from 180 bar to 250 bar until now. Before Christmas in 2023, it was found that one valve was damaged. We will continue the test for at least one more year. We sent the damaged valve to the supplier (HYDAC) in Germany for analysis. In summary, the cause of the fault is salt-like deposits that have been flushed in through the side inlet holes. The deposits led to scoring in the valve body, causing the piston to jam.



Fig.7 The field test place in Vattenfall at Älvkarleby.

### *Case 3: field test in Statkraft*

Based on the cleaning and changing knowledge gained from Svea Skog and Vattenfall. Two



field tests have been started in Statkraft: one is in Nyfors from 2023.05 until now and another one in Skeen from 2023.09. The people at Statkraft are quite satisfied and happy with the performance until now. Not any issue has been found. We will continue the test for at least one more year.



Fig. 8 The field test in Statkraft, Nyfors from 2023.05 until now



Fig. 9 The field test in Statkraft, Skeen from 2023.09 until now

#### *Case 4: field test in Uniper*

Based on the successful field test experience in Vattenfall and Statkraft. We started one field test in Verperyd Uniper in 2023.11, which is a hydraulic system for flood gate control with a tank size of 100 liters and runs under room temperature. After around one month's test, the people at Statkraft found that (1) a small part of the paint inside the hydraulic tank was damaged; (2) the pressure went up from around 80 bar to around 150 bar which goes up to their system's alarming level. After a careful discussion and investigation between LTU and Uniper, we believe that the paint damage should be due to the left cleaning fluid in the system, and the higher pressure should be caused by the lower temperature (went down to -10 °C at the middle of December from around 10 °C when started the

test). The viscosity of the hydraulic fluid was increased too much when the temperature was decreased. Based on the discussion, Uniper replaced the old tank with a new stainless tank. At the same time, we at LTU provided a new hydraulic fluid with lower viscosity at lower temperatures. We changed the hydraulic tank and the hydraulic fluid during Christmas time. It was found that the system ran very well after the change, the pressure went down from 80-150 bar to around 40 bar. Since then no technical problem has been found. The Uniper people are very happy with the situation now!



**Hydraulic system for water outlet**

Figure 10 The field test at Verperyd, Uniper.

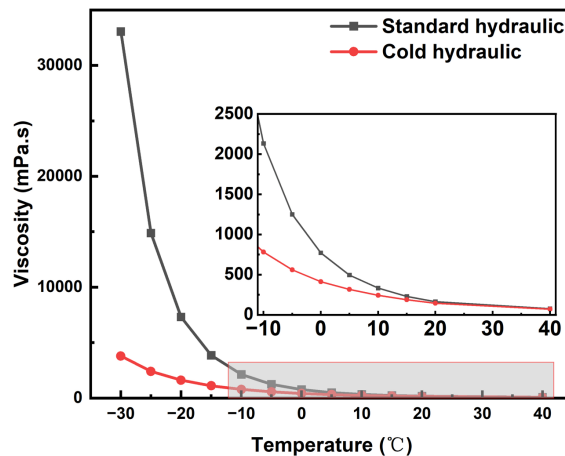
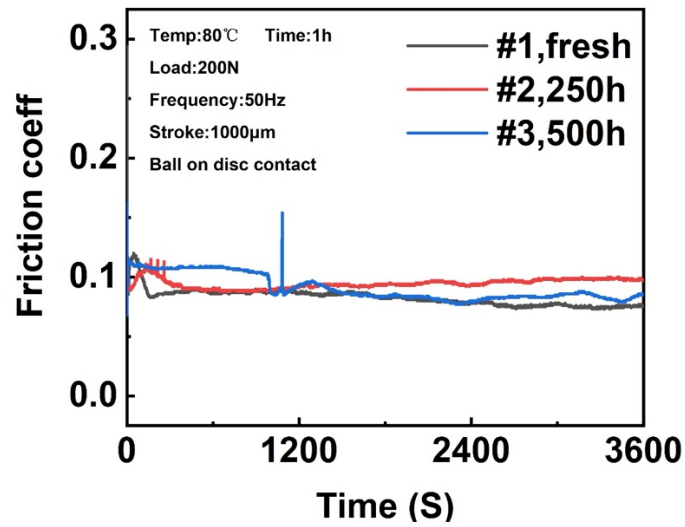


Figure 11 The viscosity of the new hydraulic fluid under different temperatures.

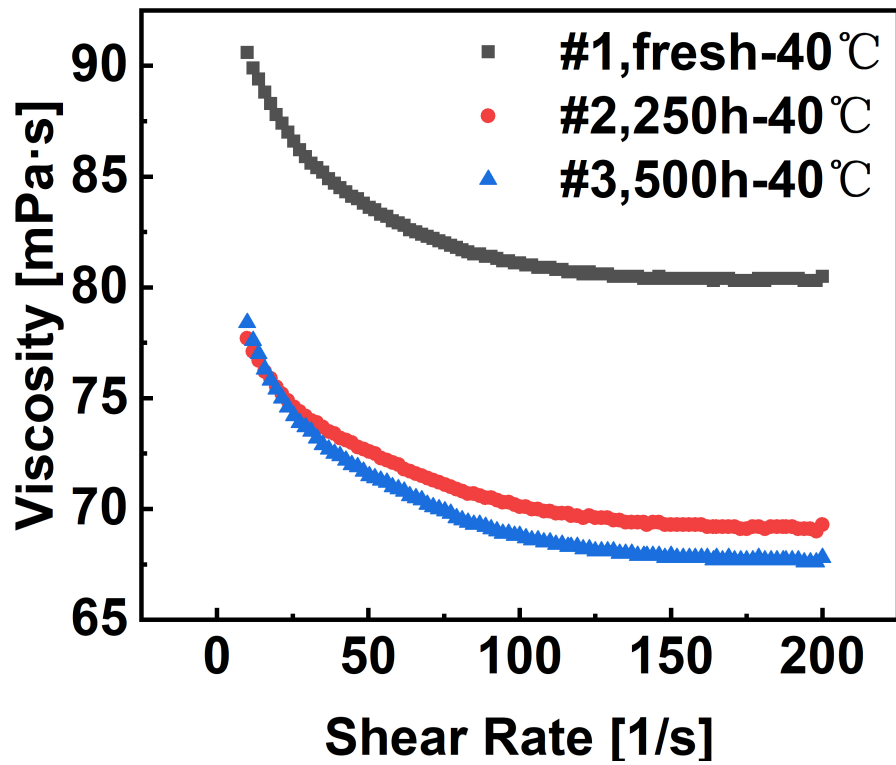
## 2. Hydraulic fluid service life study

For the field test in Svea Skog, we took out samples before summer and did some analysis.

### I. SRV friction (Figure 12)

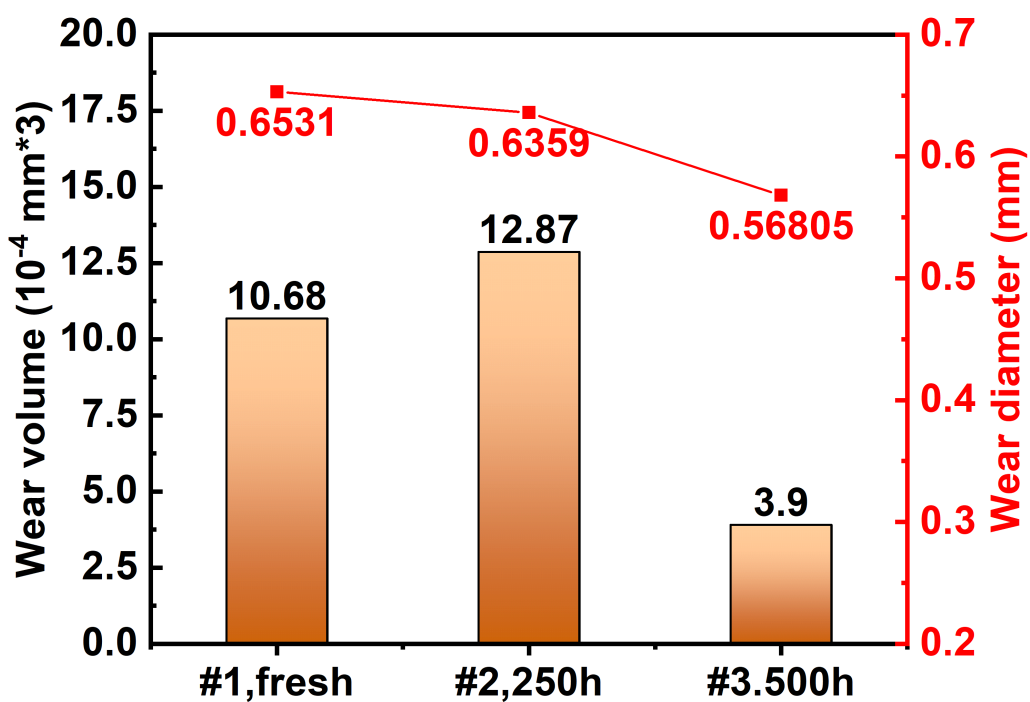


II. Rheometer (viscosity) (Figure 13)

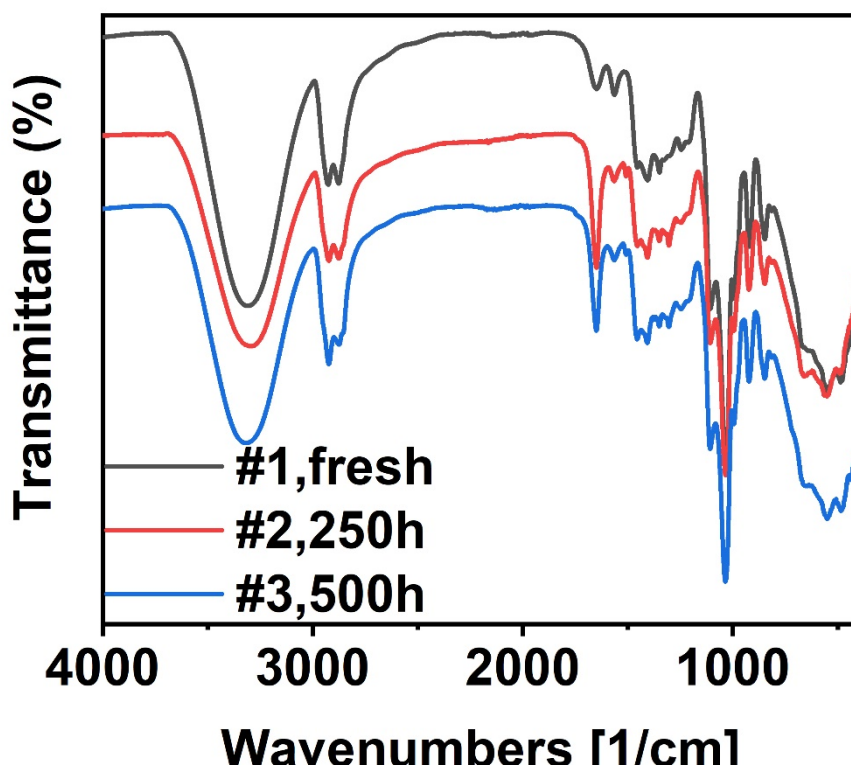


III. SRV Wear (Figure 14)

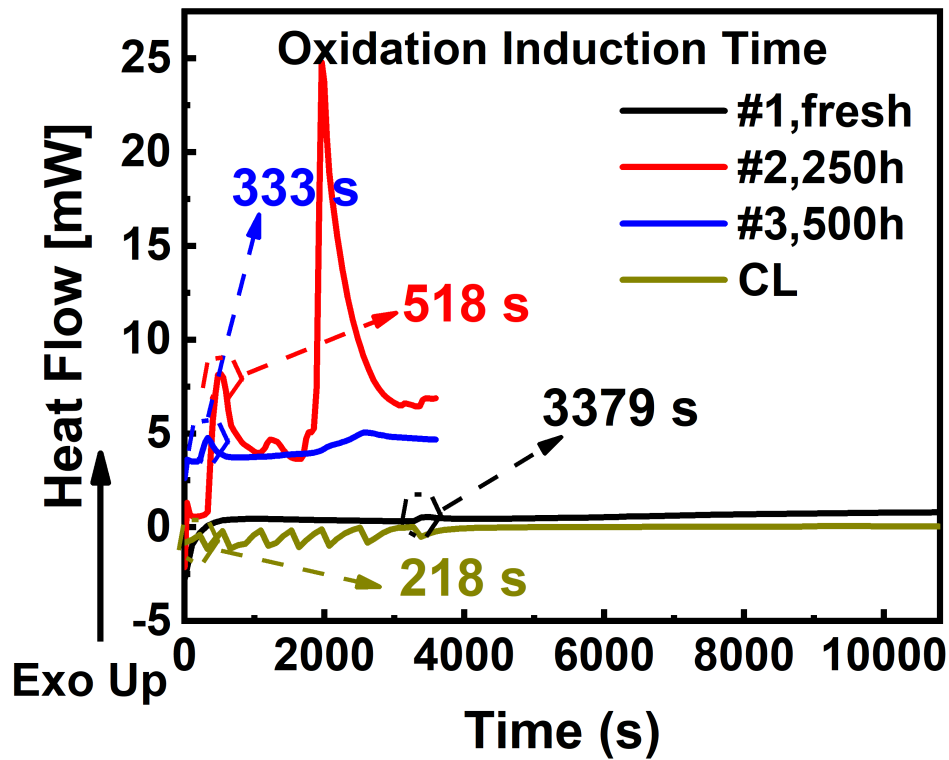




IV. FT-IR (chemical stability test) (Figure 15)



V. Antioxidation test (Figure 16)



According to the above tests, we can see that the fluid shows very good performance stability, which means that the fluid can be used for a longer time.

### 3. Quality control methods development

#### I. Quality control by measuring the density

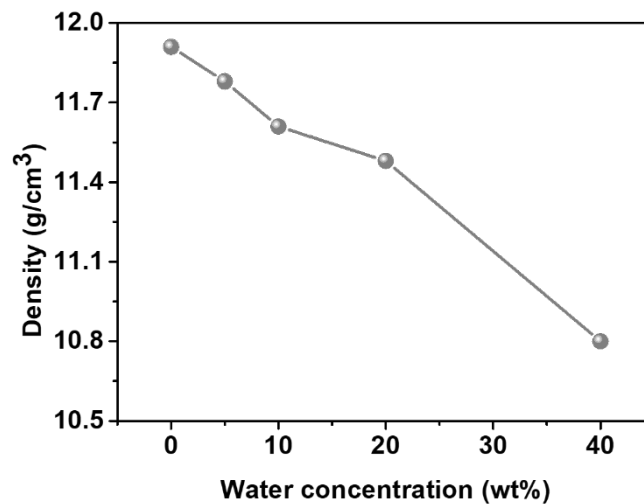


Figure 17 The density of glycerol mixed with different amount of water

It can be seen from Fig. 17 that with the increase of added water in glycerol, the density of the glycerol-water mixture is obviously decreased. Since the developed hydraulic fluid contains a certain amount of water, which is very sensitive to the fluid's viscosity. While viscosity is a super important

parameter for the function/performance of the hydraulic. So, it is possible to monitor the quality of the hydraulic fluid by monitoring its density. At the same time, it is quite convenient to measure fluid's density, which is widely applied and accepted in the industry.

## II. Quality control by measuring the electricity-generating performance

The triboelectric nanogenerator (TENG), based on the conjunction of triboelectrification and electrostatic effects, was developed for energy harvesting and self-powered in 2012. TENG-based sensors have successfully been used as mechanical sensors for detecting water waves, liquid flow rate, and organic and ion concentration based on liquid-solid contact electrification. Inspired by the works on water-based systems, a glycerol-water mixture-based hydraulic fluid condition monitoring sensor is developed (Fig. 18). The electrification process of liquid-solid contact can produce a surface charge on a large scale via electron and ion transfer on liquid-solid contact interfaces. Via the triboelectrification on the liquid-solid interface, the electric signals generated from the contact tribo-layers can detect hydraulic fluid conditions. It can be seen from Fig. 8 that with the increase in water concentration, the generated voltage is increased, which is high enough to be detected by a normal electric meter. The developed sensor is only in the dimension of  $10 \times 10 \times 3$  mm, which is quite feasible for the industry people to use. So, this developed method can also be used for quality control which can even be used for in-line monitoring purposes!

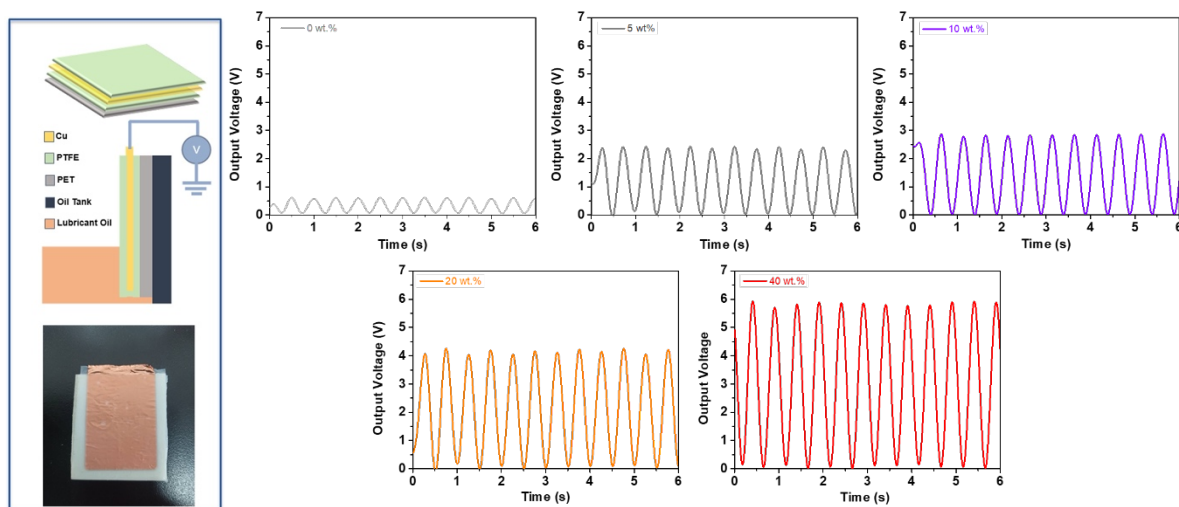
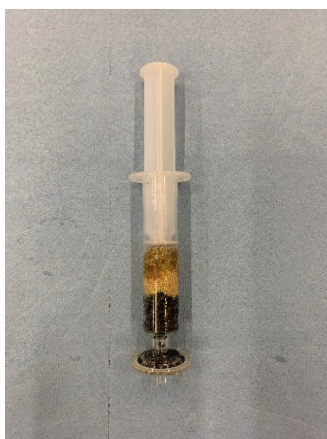


Figure 18 The TENG sensor composition and the generated voltage

## 4. Waste glycerol-based hydraulic fluid treatment.

- a. Use activated carbon, cation exchange resin, and anion resin to treat waste

## **lubricant oil**

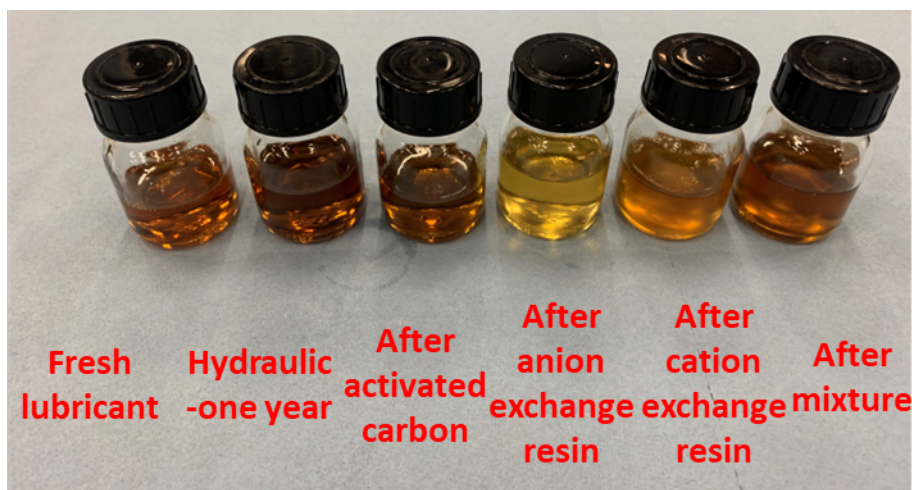


- Mix 50 ml of waste oil (Hydraulic-one year) and 10 g of activated carbon and stir at room temperature for 24 hours. Use a syringe and a 0.45µm organic filter to form a simple filtration device. As shown in the picture. Filter the treated lubricating oil - **After activated carbon.**
- Mix 50 ml of waste oil (Hydraulic-one year) and 10 g of anion exchange resin and stir at room temperature for 24 hours. Use a syringe and a 0.45µm organic filter to form a simple filtration device. Filter the treated lubricating oil - **After anion exchange resin.**
- Mix 50 ml of Hydraulic-one year with 10 g of cation exchange resin and stir at room temperature for 24 hours. Then, use a syringe and a 0.45µm organic filter to create a simple filtration system. Filter the treated lubricating oil - **After cation exchange resin.**
- Mix 50 ml of waste oil (Hydraulic-one year) and 5 g of anion exchange resin, 5 g of activated carbon and 5 g of cation exchange resin and stir at room temperature for 24 hours. Use a syringe and a 0.45µm organic filter to form a simple filtration device. Filter the treated lubricating oil - **After mixture.**



### **b. Visual observation**

Observe with the naked eye the colour changes of each oil before and after treatment.



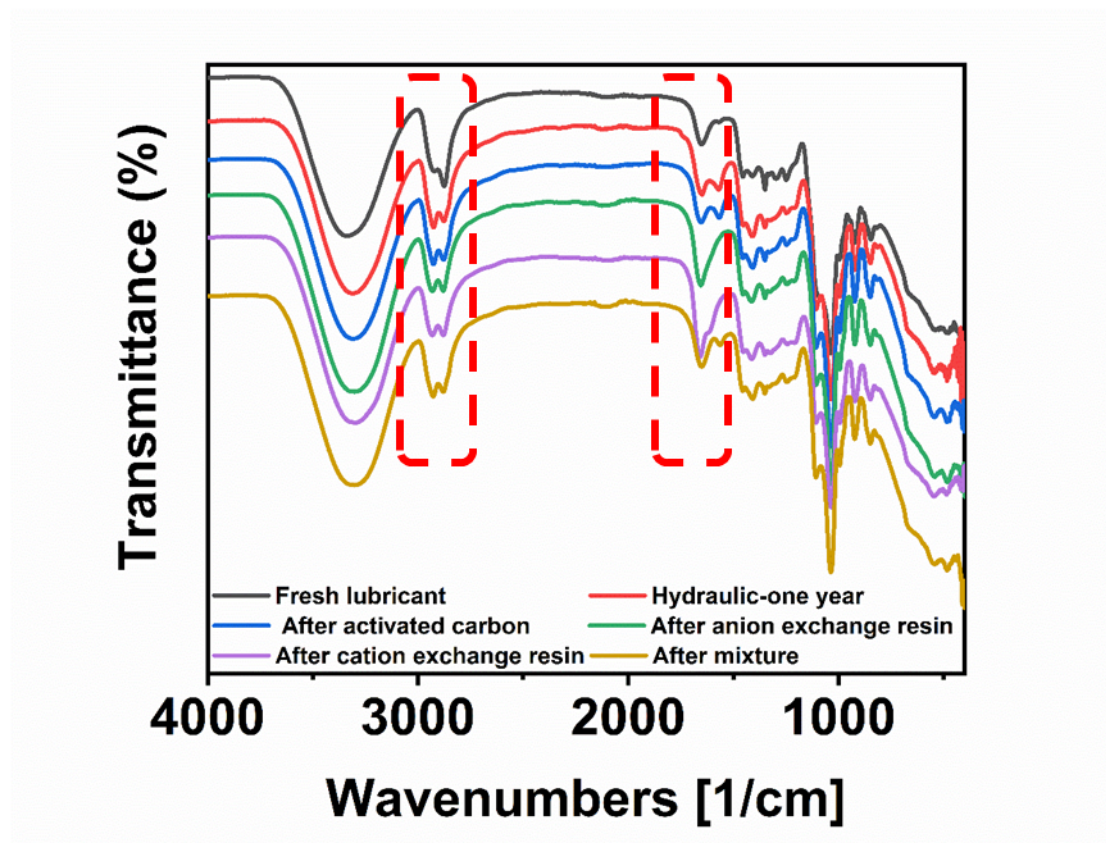
From left to right: Fresh lubricant, Hydraulic-one year, After activated carbon, After anion exchange resin, After cation exchange resin and After mixture

The initial waste oil is "Hydraulic-one year". Its colour becomes darker because some metal ions are free after use or exist in the waste oil in the form of small metal particles. After activated carbon treatment, the colour of the waste oil is almost like that of the fresh oil, indicating that the activated carbon successfully adsorbed certain components in the waste oil and reduced the depth of the colour. After being treated with anion exchange resin, the colour of the waste oil becomes lighter to yellow, which may be due to the removal of some darker additives by the anion exchange resin. After cation exchange resin and mixing treatment, the colour of the waste oil becomes lighter and closer to the original new lubricating oil. Its dues to cation exchange resin remove some metal ions.

**c. Fourier Transform Infrared Spectroscopy**

Fourier transform infrared—attenuated total reflectance (FTIR-ATR) spectra was recorded using a Nicolet Summit FTIR spectrometer. The number of scans is 16, and the resolution is 4 cm.





The

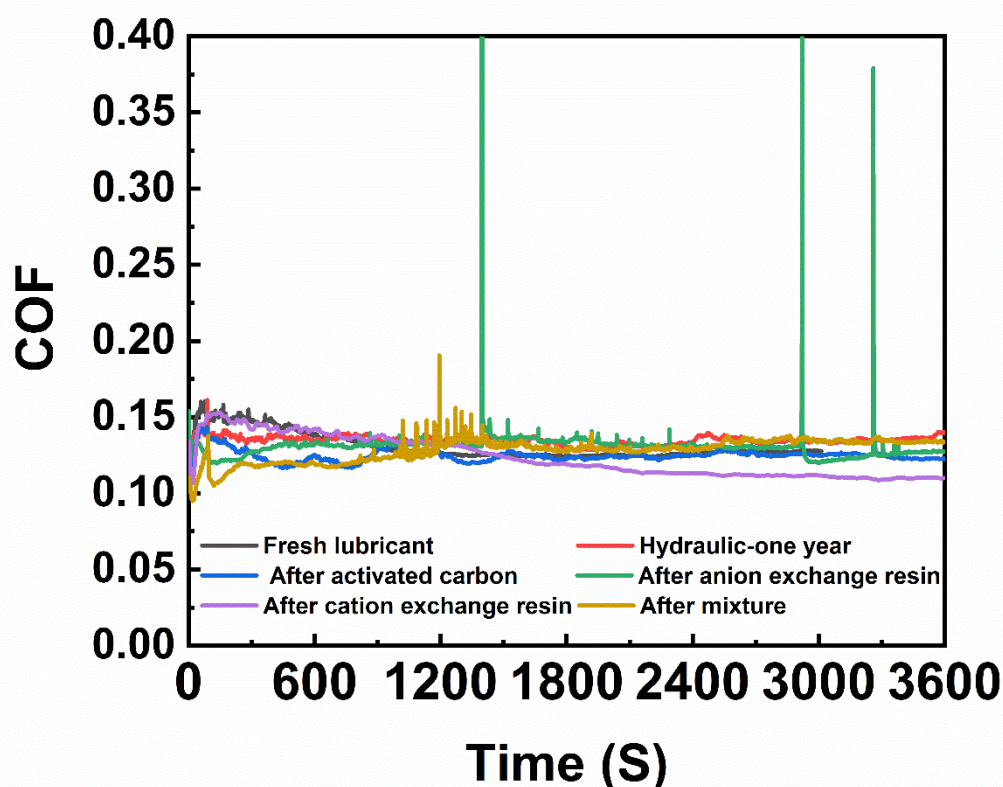
wavenumber range was determined to be 4000–400  $\text{cm}^{-1}$

Use FTIR spectrometer to observe the changes in Hydraulic-one-year waste oil and Fresh oil. A weakening of the signal at 2880  $\text{cm}^{-1}$  and an enhancement of the signal at 1569  $\text{cm}^{-1}$  were observed. This indicates that the hydrocarbons in the waste oil may have been degraded or consumed during use, and new compounds may also exist. This may be due to oxidation or other oxidation caused by the contact of the waste oil with oxygen or moisture in the external environment reaction. There was no obvious change in the infrared spectrum of oil lubricants treated with activated carbon and mixture. For lubricating oil treated with anion exchange resin and cation exchange resin, the peak at 1569  $\text{cm}^{-1}$  almost disappeared, which shows that the exchange resin has absorbed some small molecular substances in the waste oil.

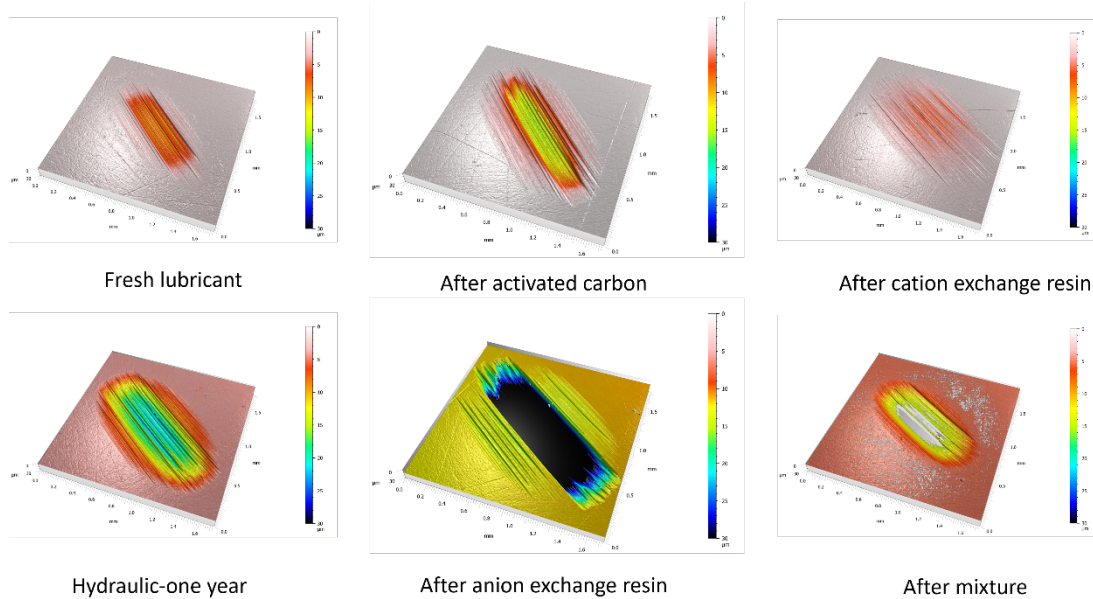
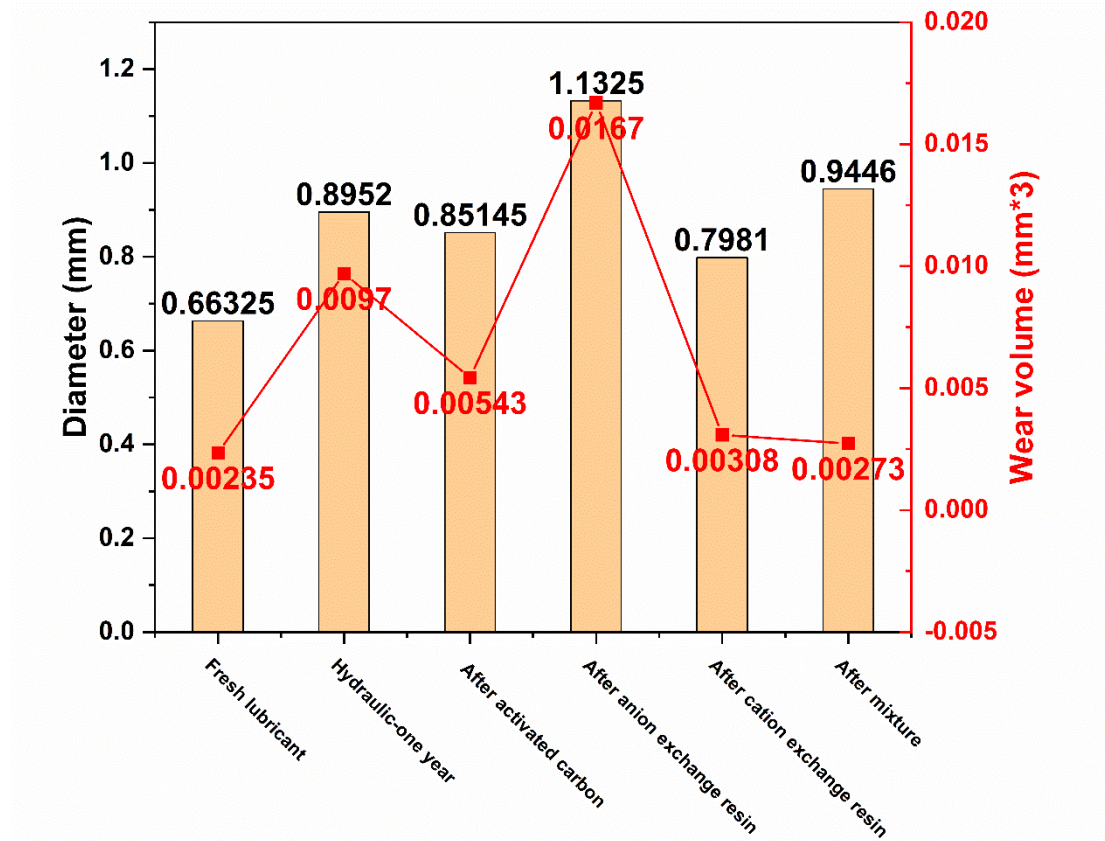
#### d. Tribology Study

To ensure the standardization of the lubricating performance tests, the parameters used in this study conformed to the ASTM D5707-11 standard. The tests were conducted on an SRV-5 tribometer (Optimol Instruments, Munich, Germany) with an upper steel ball (52 100 bearing steel, diameter 10 mm, surface roughness (Ra) 20 nm) undergoing reciprocating motion against a stationary steel disk (100CR6 ESU hardened, Ø24 mm × 7.9 mm, surface roughness (Ra) 120 nm). The tests were run for a duration of 1 h under a contact pressure of 2.72 GPa at a temperature of 80 °C, with a sliding frequency of 50 Hz and 1 mm amplitude. The experiment was repeated three times to ensure the reproducibility of the results. The lower disk wear volume loss and the upper ball worn diameter were determined by using a 3D optical profile meter (Zygo 7300) after the tribology tests.





The lubrication performance of "Hydraulic-one year" waste oil decreases significantly after one year of use. This may be due to the gradual degradation and consumption of the lubricant added to the oil or the accumulation of contaminants due to long-term use, resulting in a decrease in its lubrication performance. After activated carbon treatment, the lubrication properties of waste oil were improved. This may be because activated carbon can adsorb and remove certain impurities, pollutants, and degradation products in waste oil, thereby purifying the oil and improving its lubrication performance. After anion exchange resin and mixing treatment, the lubrication performance of the waste oil was improved, but the stability was poor. Anion exchange resins may be able to remove some negatively charged ions or molecules, further purifying the waste oil. These treatment steps may help improve the lubricating properties of waste oil and reduce friction and wear of mechanical components. Finally, after treatment with cation exchange resin, the lubrication properties of the waste oil were optimal. Cation exchange resin can remove some positively charged metal ions or small molecules, further purifying the waste oil. In addition, cation exchange resin may also be able to introduce some components that are beneficial to lubrication properties, thereby improving the lubrication effect of waste oil. Blended processing combines methods to further improve the quality and performance of waste oil. At the same time, the four treatment methods mainly use the principle of adsorption. The adsorption effect reduces the viscosity of the oil and reduces friction. In summary, the lubrication properties of waste oil are improved through activated carbon, anion exchange resin and mixing treatment, while cation exchange resin treatment further improves its lubrication effect.

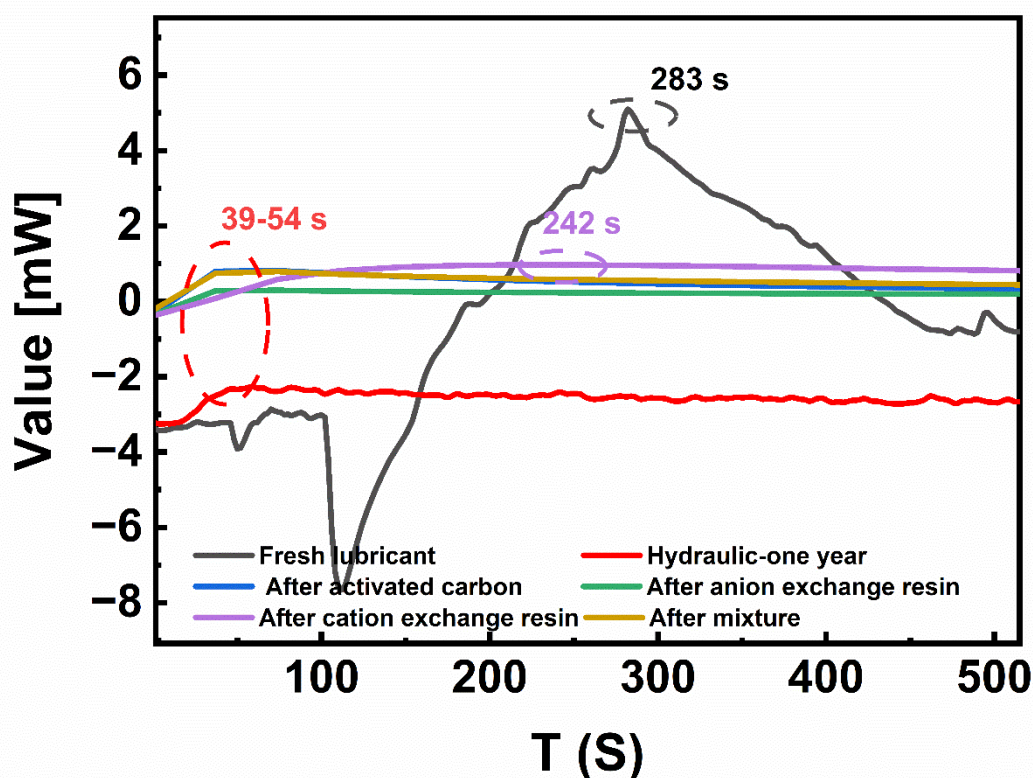


According to the figure, the anti-wear properties of waste oil changed significantly after different processing steps. The anti-wear properties of the original "Hydraulic-one year" waste oil decreased significantly after one year of use due to lubricant degradation or consumption caused by prolonged use, which increased friction and wear. After activated carbon treatment, the lubrication properties of the waste oil were improved, but the anti-wear properties were not fully restored, due to not targeting all wear mechanisms to have a significant effect. However, the anti-wear performance of waste oil after anion exchange resin and mixed treatment is worse than that of aged oil, possibly due to the removal of

some anti-wear agents during the treatment process. The anti-wear performance of waste oil treated with cation exchange resin reaches the best level, which is close to the level of fresh lubricating oil. This may be due to the successful removal of harmful components and the retention of most of the anti-wear agent components. In summary, cation exchange resin treatment is crucial to improving the anti-wear properties of waste oil, while retaining important anti-wear agent components, providing reliable technical support for the reuse of waste oil.

**e. Antioxidation Study**

Oxidation induction time (OIT) was determined by DSC (TA Instruments DSC Q2000). Samples (5–10 mg) were exposed to a nitrogen atmosphere with a nitrogen flow rate of 50 mL/min and a heating rate of 20 °C/min. After reaching the temperature of 200 °C, the samples were kept in a nitrogen atmosphere for 10 min. The nitrogen flow was then switched to an oxygen flow of 50 mL/min and the temperature was until the sample was completely oxidized. OIT was reported as the onset time of sample oxidation.



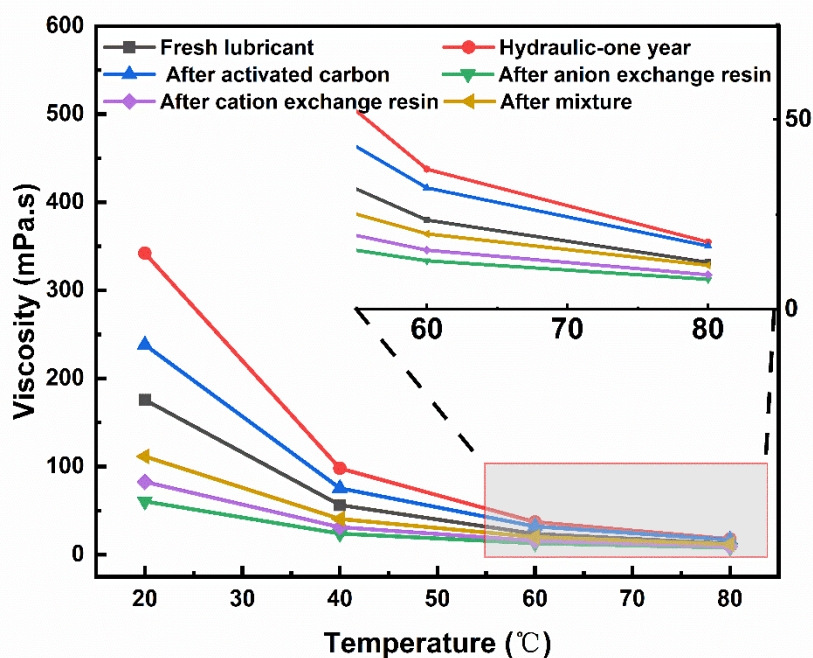
During OIT testing, changes in the antioxidant properties of waste oil were observed after different treatment steps. First, the antioxidant properties of the original "Hydraulic-one year" waste oil are significantly reduced. This may be due to the gradual degradation or consumption of the antioxidants added to the oil due to long-term use, and the introduction of new small molecules that are easily oxidized. substances, leading to a decrease in the oil's resistance to oxidation. The antioxidant performance of the waste oil that has been treated with activated carbon, anion exchange resin and mixing has not been improved, and the oxidation induction time is still in the range of 39-54 seconds. This may be because these treatments do not effectively repair the degradation or depletion of



antioxidants in the oil and do not remove small molecules prone to oxidation. However, the antioxidant performance of waste oil treated with cation exchange resin is significantly improved, approaching the level of fresh lubricating oil, and the oxidation induction time exceeds 200 seconds. This shows that the cation exchange resin successfully adsorbs and removes small molecules that are easily oxidized, improves the oil's resistance to oxidation, and makes its antioxidant performance close to the level of fresh lubricating oil. In summary, cation exchange resin treatment is an effective waste oil treatment method. It can not only improve the antioxidant properties of waste oil, but also protect or repair the antioxidants in the oil, extend its service life, and provide opportunities for reuse. Provided important technical support.

#### **f. Rheology Study**

Rheology experiments were carried out by using an Anton Paar rheometer, SmartPave 92, with a plate-plate configuration (20 mm diameter, 1 mm gap). The test temperature is 20, 40, 60, 80°C.



The viscosity changes of the oil after different processing steps were observed by Rheometer testing. "Hydraulic-one year" used oil has an increased viscosity compared to fresh lubricating oil. This may be due to the degradation or precipitation of certain components in the waste oil due to long-term use, increasing the viscosity of the oil. After activated carbon treatment, the viscosity of the waste oil is slightly reduced. The adsorption effect of activated carbon may help to remove some polymer compounds or sediments in the waste oil, thereby reducing the viscosity of the oil. However, the viscosity of waste oil after anion exchange resin, cation exchange resin and mixing treatment is significantly reduced and is much lower than fresh lubricating oil. This may be because these treatment methods successfully remove polymer compounds, sediments or other components that cause viscosity

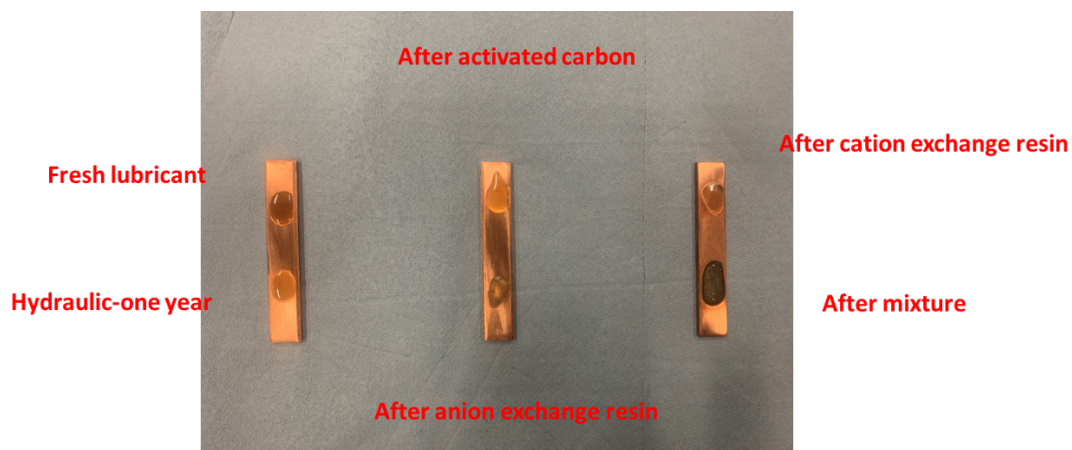
to rise in the waste oil, thereby improving the fluidity of the oil.

**g. Copper Strip Corrosion Tests**

All oils were evaluated by the ASTM D130 standard method. In a typical copper strip corrosion experiment, a strip of freshly polished and cleaned copper was dropped with one-point lubricants at 100 °C. After 24 h, the copper strips were immediately washed with acetone and ethanol. Subsequently, these copper strips were compared with the corrosion standard tint board.



Before hotting



After hotting



compared with the corrosion standard tint board

The results of copper sheet corrosion experiments conducted according to the ASTM D130 standard provide the basis for a detailed analysis of the anti-corrosion properties of oils under different treatment methods. In experiments, the copper sheet corrosion test is a commonly used method to evaluate the anti-corrosion performance of lubricating oils. By observing the effect of oil on copper corrosion, we can understand the oil's ability to protect metal parts during use. First, fresh lubricating oil, one-year-old hydraulic oil and oil treated with anion exchange resin were rated as level 3a, indicating that they have a low degree of corrosion on copper sheets under experimental conditions and have better anti-corrosion properties. These results indicate that anion exchange resin treatment may successfully remove harmful components from the oil or introduce some beneficial components, thereby improving the anti-corrosion properties of the oil. However, oils treated with activated carbon, cation exchange resin, and mixed are rated as level 3b, and their anti-corrosion properties are slightly inferior to the former. This may be due to a failure to completely remove or neutralize corrosive substances in the oil during treatment, resulting in minor corrosion. This suggests that these treatments may need further improvement to increase their effectiveness in removing harmful components and protecting metal components.

#### h. Metal concentration test

It can be seen from the below table that the metal concentration in the used glycerol-based hydraulic fluid can be effectively reduced by using ion exchange resin. The most effective is the anion exchange resin. Activated carbon has some effect, but is not so effective. While it is not a good idea to mix these 3 treating materials together.

Sample	Br ( $\mu\text{g/kg}$ )	Cr ( $\mu\text{g/kg}$ )	Cu ( $\mu\text{g/kg}$ )	Fe( $\mu\text{g/kg}$ )	Pb( $\mu\text{g/kg}$ )	Zn( $\mu\text{g/kg}$ )
Fresh sample	500	4.3	100	410	2.2	100



1 year using in Vattenfall	2336.8	29.8	2800.8	3560.9	998.6	2860.9
After activated carbon	190.2	28.3	2681.1	2596.2	942.2	1858.6
After anion exchange resin	2116.7	7.9	33.5	121.3	17.6	40.6
After cation exchange resin	750.8	10.1	442.1	1386.2	48.3	119.6
After mixture	40.6	27.2	2398.6	3368.2	920.6	2649.8