

Entry to the Stockholm Junior Water Prize 2024

**The Development of an Innovative Demulsifier  
for the Separation of an Emulsion of  
Water-Soluble Cutting Oils**

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## **I. Abstract:**

Though water-soluble cutting oil is an essential material for metal-cutting processes, its release into the water system prompts significant contaminations. Since the emulsion of water and oil cannot be completely separated, it is difficult to recycle and often designated as waste for incineration. To solve this problem, I developed a more effective demulsifier using pine powder with a lignin content of more than 50%. When pine powder and sulfuric acid were added to 5% water-soluble cutting oil, the emulsion removal effect was confirmed. At first, the process took 3 weeks at room temperature, but it was later reduced to 1 hour at 80°C. To identify the demulsifying component of pine powder, I conducted an experiment using pine extract, confirming that lignin is a demulsifying component. Then, by comparing calcium lignosulfonate to sodium lignosulfonate, I confirmed that both substances had a high demulsifying effect, yet calcium lignosulfonate showed a greater demulsification performance. As such, I confirmed that treating 200 ml of water-soluble cutting oil emulsion with 0.016 g of calcium lignosulfonate and sulfuric acid (0.048 M) removes emulsion by nearly 100%. Also, I used the activated carbons to completely eliminate the yellow colorings from the separated water layer. The analysis has demonstrated that the separated layer contains 0.006mg/L of phenols, while no other harmful substances are detected, making the aqueous layer safe to discharge. Lastly, I applied this demulsification method to two types of waste cutting fluids, which have been stored for 1 day and 3 months after the use, respectively. Although the color of the separated water layer was more brown than the control group, the separation of emulsion was successful, highlighting its potential application to real-life settings. Moreover, I performed a bottle test using calcium lignosulfonate on the artificial emulsion made by combining crude oil and surfactant (Tween 20). The lignosulfonate showed a superior demulsifying effect compared to the control group, signifying that my demulsification method can be used not only for purifying water-soluble cutting oil but also as a demulsifier in various fields, including the crude oil industry.

## **II. Key Words:**

Demulsifier, Emulsion, Water-Soluble Cutting Oil, Cutting Oil, Waste Cutting Oil, Lignin, Sodium Lignosulfonate, Calcium Lignosulfonate, Sulfuric Acid, Separation, Pine Tree Powder, Pine Tree Extract, Activated Carbon, Crude Oil

### III. Abbreviations and Acronyms:

COD: Chemical Oxygen Demand	BOD: Biochemical Oxygen Demand
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### IV. Acknowledgements:

I would like to express my sincere gratitude to Ms. Julie MacDonald, a science teacher from Chadwick International, who provided invaluable guidance and feedback throughout the research process. Also, I would like to give a special thanks to Korea Water Forum (KWF) for this opportunity to develop my research project through a series of incubation sessions and participate in the prestigious Stockholm Junior Water Prize 2024.

### V. Biography:

Hyunseung (Henry) Lee is a rising Junior at Chadwick International School located at Songdo, South Korea. He is a passionate explorer of water purification issues and marine ecosystems, pursuing his interests in scientific discoveries through participating in various international competitions. As a representative of South Korea, Hyunseung (Henry) Lee won a gold medal at the Korea Science & Engineering Fair ('23) and participated in the International Science & Engineering Fair as an environmental engineering department.

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**\*\*Unless otherwise noted, all visuals and graphs were made by the student researcher.**

## **1. Introduction:**

### **1.1 Water Soluble Cutting Oil:**

Water-soluble cutting oil, a type of lubricant used by mixing with water, is generally used in metal working and cutting processes, and is especially useful when solubility in water is required. This type of cutting oil offers high productivity by acting as a lubricant in metal removal processes and is widely used in the current metal cutting industry. The main advantage of water-soluble cutting oil is its excellent cooling effect. Since the specific heat of water-soluble



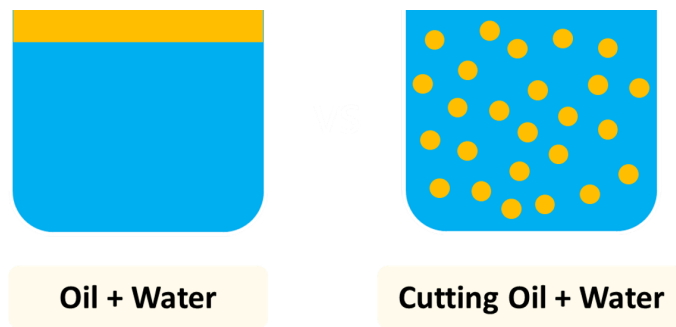
cutting oil is more than twice that of non-water-soluble cutting oil, it is more effective when cooling the heat generated during the alloy cutting process. As water-soluble cutting oil is water-based, it also offers the advantage of being non-flammable [1]. Owing to these advantages, the global metalworking oil market was valued at approximately 2.4 billion USD in 2021 and the market is expected to grow at an average of 4.2% per year to surpass 3.5 billion USD by 2030 [2].



*Figure 1. Demonstration of how water soluble cutting oil is used in metal cutting industries, including the cutting fluids tank (left), cutting fluids during metal cutting process (middle), and cutting fluids before the use (right).*

## **1.2. Problems:**

Currently, the widespread use of water soluble cutting oil is followed by the generation of more than 2 billion tons of waste-cutting oils every single year, which is enough to fill the entire city of Stockholm with a tsunami of 75 m height [3]. The use of cutting fluids is prevalent in both economically developed and underdeveloped countries, illustrating how the generation of waste cutting fluids is a global issue. However, as shown in Figure 2, unlike regular oil that forms a layer on the surface of water, water-soluble cutting oil mixes readily with water; the waste cutting fluids usually have the COD higher than 100,000mg/L and BOD over 10,000mg/L due its emulsified state [3]. Therefore, discharged water-soluble cutting oil can cause the spread of toxic compounds throughout water bodies, polluting water sources and causing destruction of marine flora and fauna ecosystems. As extremely small amounts of water soluble cutting oils added to the lake, river, or ocean can contaminate the entire water body, water soluble cutting oil is a significant international water issue.



*Figure 2. Illustration of (left) typical oil and water mixture that are immiscible and (right) cutting oil and water mixture with emulsions. Orange color indicates oil and yellow color indicates water.*

Furthermore, the purification of emulsions, consisting of 10 percent of water soluble cutting oil and 90 percent of water, involves the separation of oil from water. This process requires high thermal energy - about 800 °C of heat - leading to serious carbon emission and environmental pollution. Thus, incinerating the waste cutting oil is an environmentally devastating and energy-inefficient process. One other solution to the treatment of waste cutting oils is separating the oil from water through breaking the emulsions. This process is also called a demulsification, and current metal-cutting industries utilize various demulsification methods to treat the waste cutting oils.

### **1.3. Literature Review:**

The methods currently used for demulsification of water-soluble cutting oil can be divided into three main types. First, treating emulsions with chlorides of calcium, magnesium, and aluminum can destabilize the emulsion and lead to agglomeration of water droplets. This method is referred to as demulsification using inorganic salts, which is a prospectively effective and inexpensive method. However, the effectiveness of inorganic salt may vary depending on the type, concentration, and pH of the salts, which presents the inconvenience of having to choose the right method for each situation. Moreover, when inorganic salts are released into nature, they can increase water and soil salinity and produce toxic substances, posing threats to the environment. Also, inorganic salts are often used to separate water from the extracted petroleum, but they are widely ineffective for artificially created emulsions using emulsifiers. Thus, inorganic salt could not completely separate the water soluble cutting oils [4].

Secondly, microorganisms are used to naturally decompose cutting oil. When cutting oil is decomposed by microorganisms, a layer is formed between the oil and water and the water

droplets agglomerate, enabling purification. Since no additional chemicals are added in this process and the purification process is driven mainly by microorganisms, this method causes less environmental pollution. However, the scope of application is limited to only biodegradable cutting oil. Moreover, this approach is much less efficient than conventional methods, and contamination during degradation by microorganisms can also cause malodor and corrosion and suffer from performance degradation. Thus, microbial demulsification is also much more time consuming, and also requires a significant amount of energy [5].

Lastly, the use of electrocoagulation with an electric current can destabilize emulsions to separate cutting oil from water. Here, a redox reaction is triggered, forming agglomerates and precipitates, facilitating the removal of oil by methods such as filtration. As an advantage, the treated water can be discharged and the oil can be recycled for reuse. However, this method has the disadvantages of high installation and maintenance costs and significant electrical energy cost. In addition to the cost factor, there is also the environmental issue associated with the waste generated after electrocoagulation [6].

As such, currently available cutting oil purification methods do not simultaneously satisfy the requirement for both excellent performance and eco-friendliness. While demulsification methods with high productivity often pose harms to the environment, the eco-friendly methods generally tend to have poor performance and increased cost. However, most importantly, none of the existing demulsification methods can not achieve a near 100% separation rate of oil and water from the emulsion. Once the emulsion is completely separated, the COD and BOD of the solution drops drastically, and the environmental threats of water soluble cutting oils can be alleviated. Thus, the demulsifier for complete separation of oil and water is essential for the treatment of waste cutting fluids.

#### **1.4. Objective:**

The primary objective of this research is centered around developing a demulsification method for water soluble cutting oil with near 100% separation ability of oil and water. After the complete separation of the emulsion, the water layer should be discharged without any potential harm to the environment, while the remaining oil layer can either be reused or burned as a fuel.

## **2. Materials and Methods:**

### **2.1. Preparation of pine tree powder and wood materials:**

1. Pine tree powder (with a lignin content of  $\geq 50\%$ ) from the microbial degradation of cellulose and hemicellulose was prepared. By heating at  $200\text{ }^{\circ}\text{C}$  for 10 min, pine tree powder samples with increased hydrophobicity and lipophilicity were prepared.
2. Pine and fir trees were ground to prepare wood chips with a size of  $\leq 3\text{ mm}$ .

### **2.2. Demulsification of water-soluble cutting oil emulsion:**

1. Water-soluble cutting oil solution (5%) was prepared by adding 10 ml of water-soluble cutting oil (SY-100, Samyuk Special Oil Solution Co., Ltd.) to 190 ml of distilled water.
2. After adding 200 ml of the 5% water-soluble cutting oil to a 600 ml beaker, a certain amount of 95% sulfuric acid (Daejung) was added and mixed.
3. The pre-prepared pine tree powder (10 g) was added to the beaker, either in powder form or as powder in a tea bag.
4. The demulsification effect was observed at various temperatures and the transmittance (%T) at 600 nm was measured and recorded.

### **2.3. Pine tree powder extract:**

1. After using pine tree powder to demulsify water-soluble cutting oil (SY-100), an aqueous solution layer was prepared by filtering with filter paper.
2. Using distilled water as a solvent, pine tree powder was added to a 1 L flask and an autoclave was used for extraction at  $121\text{ }^{\circ}\text{C}$  for 1 h.

### **2.4. Demulsification effect of pine tree extract:**

1. Using varying concentrations of the extract, the demulsification effect of the pine tree extract on 5% water-soluble cutting oil was measured with variation of the pH, temperature, and time.
2. The transmittance (%T) was measured and graphically plotted to determine the degree and rate of separation.

### **2.5. Preparation of lignosulfonate samples:**

1. Calcium lignosulfonate and sodium lignosulfonate were purchased from Daemyung Chemical.

#### **2.6. Demulsification effect of lignin:**

1. The water-soluble cutting oil purification effect of calcium lignosulfonate and sodium lignosulfonate was investigated.
2. For this purpose, the same 5% water-soluble cutting oil was used and the degree of separation was measured while adjusting the amount of lignin, pH, and time.
3. The transmittance (%T) was measured and graphically plotted to determine the degree and rate of separation.

#### **2.7. Activated Carbons for Purification of Aqueous Layer**

1. Activated Carbons (Shin Kwang Chemical) are prepared to purify the separated water layer from the cutting fluids.
2. Three different types of activated carbons, including 40 mesh, 60 mesh, 200 mesh, are used, and the solution was treated under a magnetic stirrer for 4 hours, followed by the centrifugation process.

#### **2.8. Water quality analysis of purified aqueous solution:**

1. The final separated aqueous layer of the solution was sent to the Seoul National University National Instrumentation Center for Environmental Management (NICEM) for more accurate water quality analysis and to determine whether there are any remaining substances that may cause harm to the environment.

#### **2.9. Demulsification of Waste Cutting Fluids**

1. Obtained two types of waste cutting oils from Hosung Precision (Incheon, Korea); the first waste cutting fluid has passed 1 day after its use, and the second waste cutting fluid has passed 3 months after its use.
2. Emulsion of waste cutting fluids has been separated using the calcium lignosulfonate and sulfuric acid, following the same method as above.

#### **2.10. Demulsification effect on crude oil:**

1. By adding 175 ml of distilled water, 5 ml of Tween-20, and 20 ml of crude oil (supplied by Eil Industrial Laboratory) to a 200 ml beaker, an emulsion was formed by vigorously stirring at 60 °C.
2. After adding 22 ml of the prepared emulsion to a test tube, the experimental group was treated with calcium lignosulfonate (0.05 g/L) and the demulsification effect was examined.
3. A test tube was used to conduct a bottle test and the test tube height was used to measure the volume of the aqueous layer (22 ml) to determine the demulsification efficiency.

### **3. Results and Discussion:**

#### **3.1. Effect of Pine Tree Powder and Sulfuric Acid on Demulsification:**

From the previous projects, I created a pine tree powder, which is an eco-friendly oil absorbent created using pine trees with increased lignin content. Due to its hydrophobic nature, pine tree powder is effective when absorbing oils on the surface of water bodies. Thus, I first attempted to demulsify the water soluble cutting oils using the pine tree powder due to its renowned excellence as an oil absorbent. First, I treated the water soluble cutting oils using only the pine trees, but the demulsification effect did not appear. Then, through the literature review, I identified the effect of sulfuric acid on destabilizing the emulsions, and I added sulfuric acid along with the pine tree powder to observe the demulsification process of water soluble cutting oils. Although it took 2~3 weeks, the emulsion was separated in the experimental group that was treated with sulfuric acid, unlike the control group that was not treated with sulfuric acid, as shown in the figure 3. When only the sulfuric acid was added to the solution, the demulsification effect did not appear, but a thin layer of oil was formed on the surface of the emulsion. Accordingly, the demulsification effect of pine tree powder was confirmed, and at the same time, it was found that pH is an important factor affecting the demulsification of water-soluble cutting oil.

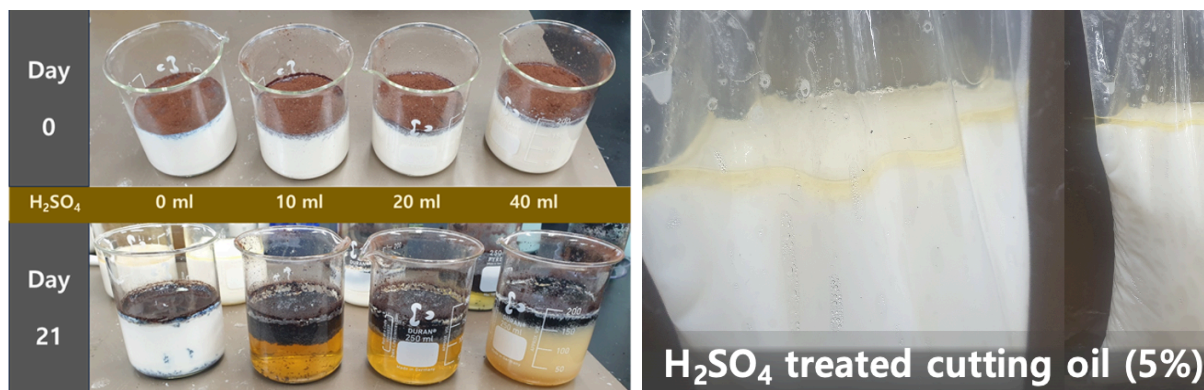


Figure 3. Demulsification effect of sulfuric acid and pine tree powder confirmed through the adding 12g of pine tree powder to 200ml of cutting fluids (5%), along with 0ml, 10ml, 20ml, and 40 ml of sulfuric acid. The left photo compares the demulsification process between Day 0 and Day 21 of the reaction, while the right photo is a separated oil layer after adding only sulfuric.

### 3.2. Effect of Temperature on Demulsification:

In the subsequent investigation, pine tree powder was used by placing it in a tea bag (PET) and the reaction temperature was set for 25°C, 60°C, and 80 °C. According to the figure 4, the time required to demulsify the emulsion was reduced from 2–3 weeks at room temperature to three days at 60 °C, and nearly 1 hour at 80 °C, signifying the importance of temperature during demulsification. However, as the demulsification effect beyond 80 °C had no significant difference, I concluded that heat treating the emulsion at 80 °C is the most effective and efficient.



Figure 4. Amount of time consumed to separate the emulsion of cutting fluids for each temperature, including 25°C, 60°C, and 80 °C, organized in a graph (left) and photo (right).

### 3.3. Separating Emulsion using Pine Tree Extract:

As the sulfuric acid forms the thin oil layer on the surface, I hypothesized that pine tree powder consistently absorbs oil on the top of the emulsion, facilitating the demulsification. However, according to the hypothesis, the demulsification process should work for any types of

oil absorbent but not only pine tree powder. Thus, I used the polypropylene oil absorbent instead of pine tree powder, along with the sulfuric acid, to test the demulsification effect.



Figure 5. The effect of pine tree extract in separating the emulsion of cutting oils, illustrated in a photo (left) and graph (right). Pine tree extract and cutting oil have three different ratios, - “extract 60” on the graph signifies that 60% of the solution is pine tree extract and remaining 40% is cutting fluids.

However, when polypropylene oil absorbent was used for demulsification instead of pine tree powder, the polypropylene oil absorbent had no effect. Therefore, it was determined that demulsification was not due to oil absorption by pine tree powder, but the presence of a demulsifier component in pine tree powder. As the experiment progressed, the aqueous layer with the oil removed turned a bright yellow color, which was determined to be due to the component extracted from the pine tree powder. Thus, the effectiveness of the aqueous layer for purifying water-soluble cutting oil was investigated. According to figure 5, the result showed that the emulsion was separated more effectively as the concentration of the extract increased. Thus, the pine tree extract generated by using water as a solvent was also confirmed to be effective for demulsification. Therefore, it was determined that a certain demulsifier component inside the water-soluble pine tree extract facilitates the separation process.

### 3.4. Demulsification effect of various tree types:

Another experiment was conducted to identify the demulsifier component in pine tree and pine tree extract with water as a solvent. Fir tree bark, pine tree bark, and waste wood powder collected from the mountain were used as the experimental groups. As shown in figure 6, all three experimental groups had a demulsification effect, but the result demonstrated that the demulsification effect was inferior to that in the previous experiment using pine tree powder.





Figure 6. The demulsification effect of various tree types, including fir tree bark, waste wood powder, pine tree pellet, and pine tree bark (same amount of sulfuric acid added for all samples).

In attempts to identify the demulsifier component in the pine tree extract, we contacted various laboratories, including the KIST Advanced Analysis Center, but the laboratories notified us that HPLC analysis could not be performed due to the difficulty in procuring the lignin standard solution analysis. Moreover, we were also notified that FTIR analysis would be difficult because the sample is an aqueous matrix. Nonetheless, based on the fact that pine tree powder with high lignin content demonstrated superior demulsification effect than other types of tree samples and all tree samples showed demulsification effect to certain extent, I postulated that the lignin, a common component among trees, may be the demulsifying agent. Also, in the previous research, I have used the Lignin Klason Method, which involves the use of sulfuric acid to obtain lignin from the wooden materials. As my demulsification process also consists of the assistance from the sulfuric acid, I draw a logical conclusion that lignin facilitates the demulsification of water soluble cutting oils. Accordingly, calcium liginosulfonate and sodium liginosulfonate were purchased from Daemyung Chemical and used in the experiment.

### 3.5. Demulsification effect of Lignin:

I used both calcium liginosulfonate and sodium liginosulfonate instead of pine tree powder to separate the emulsion of water soluble cutting oils. As shown in figure 7, both types of liginosulfonate demulsified the mixture of water and water-soluble cutting oil, where calcium liginosulfonate was found to be more effective than sodium liginosulfonate. In addition, demulsification was facilitated as the amount of liginosulfonate increased, but there was no difference in the effect beyond a certain amount. Also, the transmittance decreased at 3 hours, but it was determined that this was due to the increase in the amount of liginosulfonate.

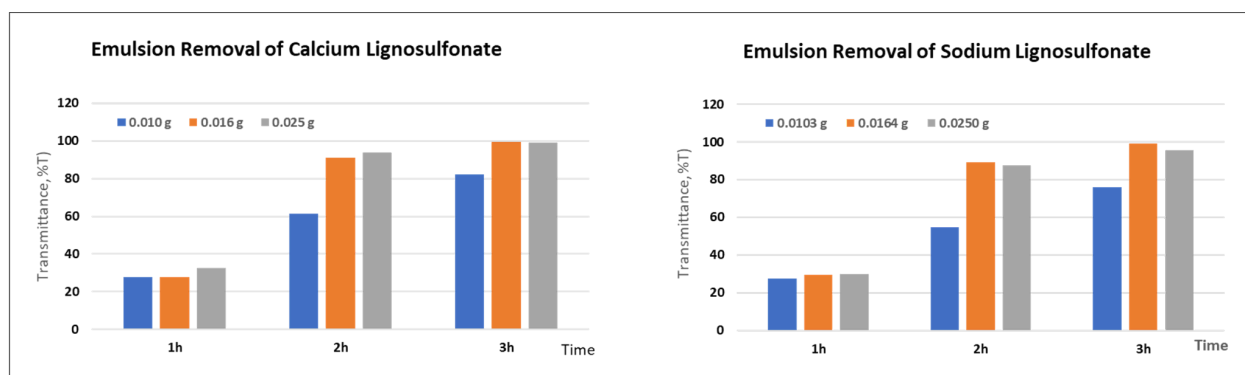


Figure 7. Demulsification effect of calcium lignosulfonate and sodium lignosulfonate visualized in graphs (0.01g, 0.016g, and 0.025g signifies the amount of lignosulfonate added to the emulsion).

The effect of the amount of sulfuric acid and lignosulfonate on demulsification was investigated. As shown in figure 8, when the concentration of sulfuric acid increased, both types of lignosulfonate showed a greater demulsification effect, whilst increasing the amount of lignosulfonate also had a greater effect on demulsification. However, the amount of lignosulfonate was more important than the amount of sulfuric acid, as the demulsification effect did not appear unless a certain amount of lignin or more was added. Most importantly, none of the previous literature has been conducted regarding the effect of lignin as a demulsifier, contributing to the novelty of my finding.

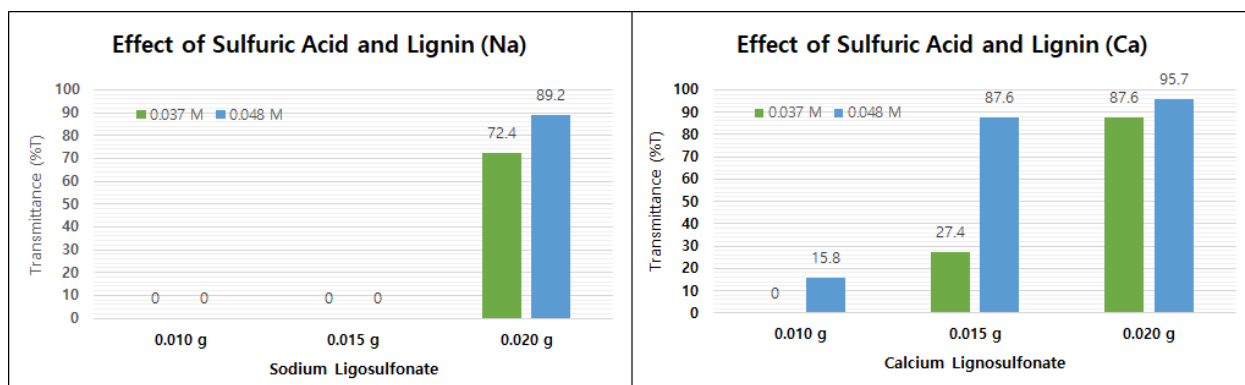


Figure 8. Effect of sulfuric acid in separating the emulsions, along with two types of lignosulfonates, visualized in graphs (0.037M and 0.048M signifies the morality of sulfuric acid added to the emulsion).

### 3.6. Purification of Separated Water Layer using Activated Carbon:

Then, I treated the separated water layer using 3 different types of activated carbon to completely eliminate the coloring. The color of the water layer derives from the pigment in the cutting fluids, varying from yellow to dark-brown color by different concentration of the cutting

oils and the amount of lignin added. As a result, the colorings present in the separated water layers were completely eliminated, and the water layer has become transparent as shown in figure 9.

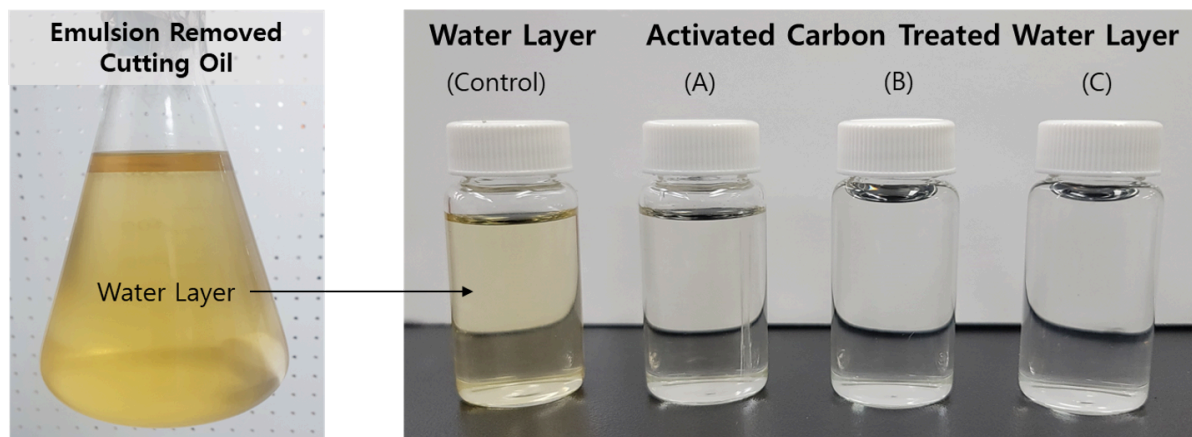


Figure 9. Effect of activated carbons in purifying the separated water layer from the emulsion, where sample A is 200 mesh, sample B is 60 mesh, and sample C is 40 mesh.

### 3.7. Safety Analysis on Separated Aqueous Layer:

To investigate the identity and quantity of the remaining oil components with potential detrimental environmental impact in the demulsified aqueous layer, twenty items were sent to Seoul National University NICEM for analysis, including phenols. According to figure 10, the results showed that phenols accounted for 0.006 mg/L, while other components were not detected. Therefore, it was determined that the demulsified aqueous layer can be discharged safely into sewage without harm to the environment.

Name	Value	Name	Value
Phenol	0.006 mg/L	Toluene	ND
Benzene	ND	Dichloromethane	ND

Figure 10. Table with amount of Phenol, Benzene, Toluene, and Dichloromethane detected in the separated water layer of the cutting fluids.

### 3.8. Application to Waste Cutting Oils:

Then, I applied my demulsification process to waste cutting oils to identify the potential application of my demulsifier in real-life settings. Unlike the water soluble cutting oils in the laboratory that only contains the mixture of oil and water, the waste cutting oils from the field

consists of various unknown substances that are added during the metal cutting processes. Thus, I demulsified the waste cutting fluids A and B, which are used in real industrial settings. While cutting fluid A is just used to cut the metal, cutting fluid B has been stored for 3 months after the use, as shown in figure 11.

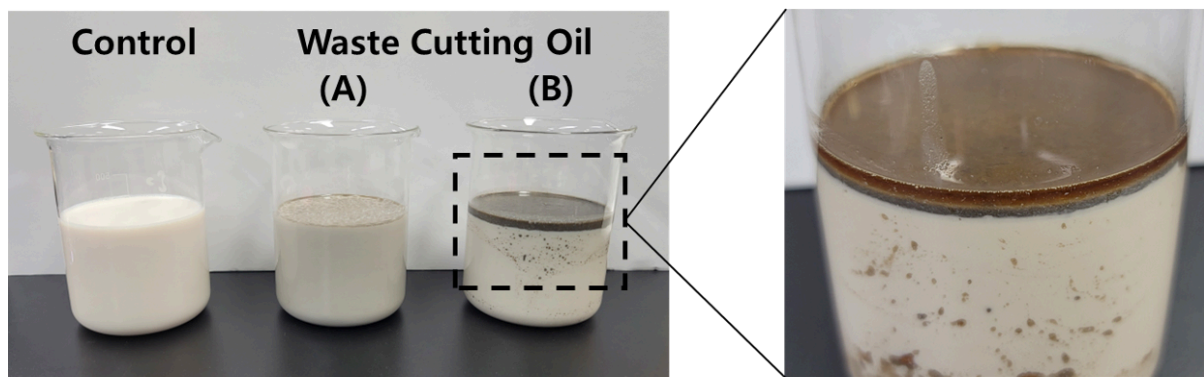


Figure 11. Cutting fluids in a laboratory setting (control), waste cutting oil A (stored for 1 day after the use), and waste cutting oil B (stored for 3 months after the use) before the separation of emulsion.

As a result, the emulsion has been separated, forming a layer between oil and water. However, the color of the water layer was more brown than the control group. Also, the microfluidic chip and foam stayed in the oil layer, leaving no additive materials to the water layer, as shown in figure 12. Thus, the water layer can be discharged after the treatment, while the remaining contents in the oil layer (less than 2~3%) can be incinerated after collecting the oils.

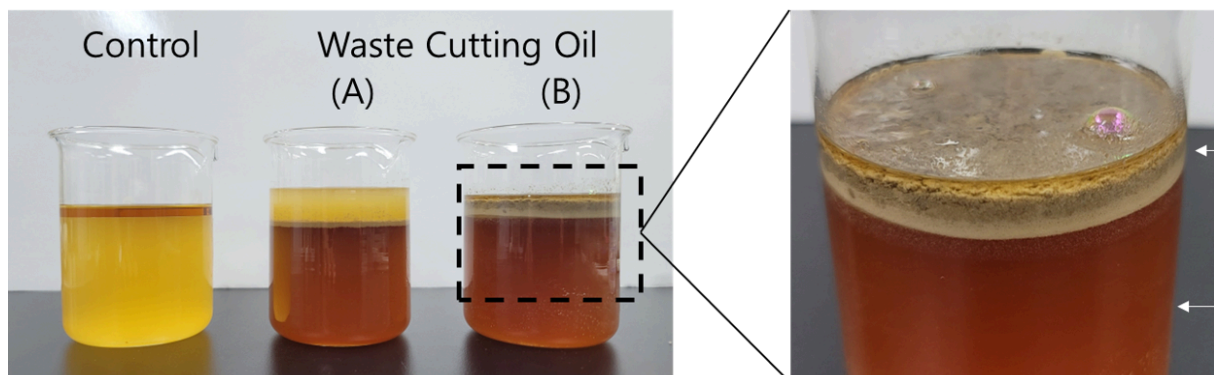


Figure 12. Cutting fluids in a laboratory setting (control), waste cutting oil A (stored for 1 day after the use), and waste cutting oil B (stored for 3 months after the use) after the separation of emulsion.

### 3.9. Demulsification Effect of Lignin on Crude Oil:

Crude oil obtained with cooperation from Eil Industrial located in Yeosu, Jeollanam-do, was used to evaluate the demulsification effect of the extract on crude oil. Tween-20, a non-ionic surfactant, was used to artificially form an emulsion by electronic stirring. The bottle test was performed at 60 °C with 0.05 g/L of calcium lignosulfonate as the final concentration.

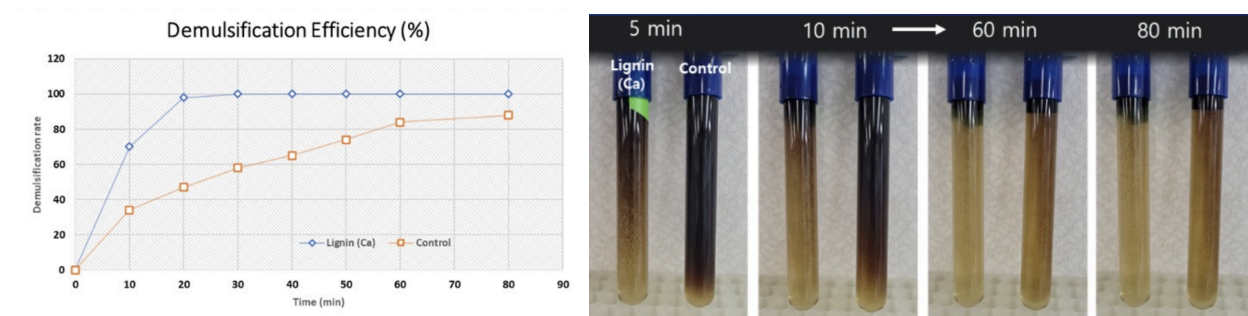


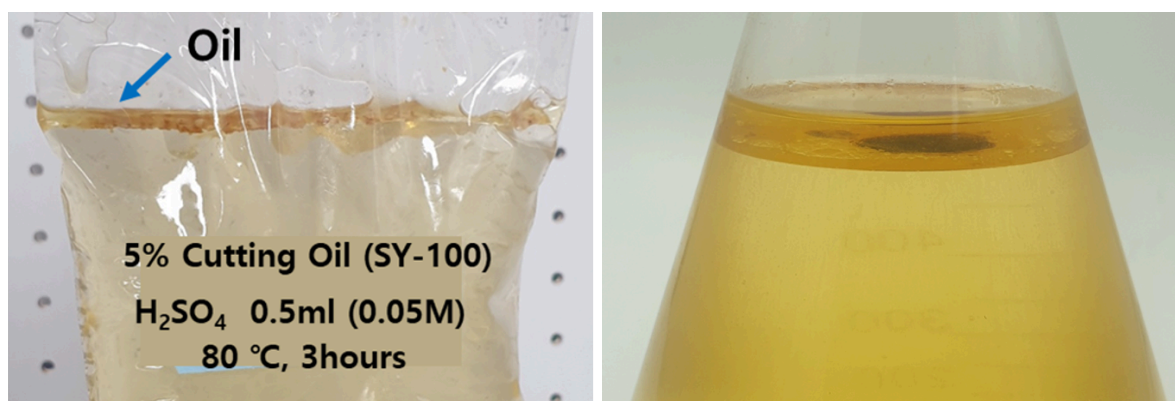
Figure 13. Demulsification effect of calcium lignosulfonate on separating the artificial emulsions created using surfactant (Tween-20) and crude oil, visualized in a graph (left) and photo (right). In the photo, the left sample is treated with calcium lignosulfonate and the right sample is not treated at all, and their separation was recorded in 5 min, 10 min, 60 min, and 80 min terms.

According to figure 13, the results showed that lignin has an excellent demulsification effect compared to the control group, and nearly 100% separation of the aqueous and crude oil layers was achieved within 20 min. In contrast, the control group showed a pattern of continuous separation of the aqueous layer, albeit at a relatively slower rate, and separation stalled at 90% even after 80 min. Therefore, the experimental results confirmed that lignin has the potential for use as a demulsifier not only for water-soluble cutting oil, but in various fields including the crude oil industry.

#### **4. Conclusion:**

In conclusion, as shown in figure 14, I succeeded in separating the water and oil from the emulsion up to near 100%, becoming the first demulsifier in the world with complete separation ability. While separated water can be discharged without harms to the environment, the oil can either be incinerated or reused.





*Figure 14. Photo of separated water layer and oil layer from the cutting fluids, where 5% cutting oil (SY-100) was treated with 0.5ml of sulfuric acid (0.05M) for 3 hours at 80 °C.*

Also, I discovered that lignin is effective for artificial emulsion created using surfactant (Tween-20) and water, posing its potential use for more real-life applications including the petroleum industry. Lastly, I used the same method to separate the waste cutting fluids into oil and water. Unlike the laboratory setting, waste cutting fluids consist of various unknown substances that are added during the metal cutting processes. Thus, demulsifying the waste cutting fluids reinforced the potential application of my demulsifier into real-life industrial settings.

In future, I aim to identify the demulsification mechanism of lignin. Also, I have to test the demulsifier with various types of waste cutting fluids to ensure that my method is applicable to industries in real-life. Finally, I attempt to reduce the amount of heat energy used to decrease the total amount of carbon emissions generated during the demulsification process.

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