

## FLOTATION OF HYDROPHOBIC MINERALS IN HALLIMOND TUBE

Aleksandra Błaszczów<sup>1</sup>, Tomasz Ratajczak<sup>1\*</sup>, Danuta Szyszka<sup>1</sup>

<sup>1</sup> Faculty of Geoengineering, Mining and Geology, Wrocław University of Science and Technology,  
Wrocław, Poland

**Abstract:** This study analyses the effect of hydrophobicity on the floatability of sulphur, silicon, fluorite and galena. Flotation was performed in a Hallimond tube in distilled water and in the presence of a frother ( $\alpha$ -terpineol). The results confirm that all the analysed minerals showed floatability in distilled water, with a varying yield, depending on the contact angle and density of each mineral. The introduction of a frother significantly improved the flotation efficiency, mainly for sulphur, silicon and fluorite.

**Keywords:** froth flotation, hydrophobic minerals, contact angle, Hallimond tube, flotometry

### 1. INTRODUCTION

Mineral processing is a key part of the global mining industry, with a significant technological and economic impact. It is a fundamental stage of the production chain aimed at obtaining valuable raw materials required by many industries. All mineral processing activities involve the separation of valuable minerals from rock materials, which poses a significant technological challenge (Gaudin, 1967; Wills, Finch, 2015; Jain et al., 2016).

Flotation is one of the main methods of mineral beneficiation, and a key element of this process is flotation equipment, commonly referred to as flotation machines. A basic laboratory-scale flotator is the Hallimond tube (Hallimond, 1944). It is a simple device, designed to test the flotation of mainly single minerals and to assess their hydrophobicity (Drzymala, 2007, 2009). The device is a vertical, usually glass tube, into which a

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\* Corresponding authors: tomasz.ratajczak@pwr.edu.pl (T. Ratajczak)  
doi: 10.37190/msc243112

mixture of mineral suspension and air is introduced. Air bubbles lift hydrophobic mineral particles towards the surface, where the mineral grains are then collected in the form of a concentrate after the gas bubble breaks at the liquid-gas interface. The Hallimond tube is widely used in fundamental research into the physical chemistry of flotation processes (Farrokhrouz and Haghi, 2009; Wills, Finch, 2015; Corpas-Martínez et al., 2020;; Hassanzadeh et al., 2021). With this apparatus, it is possible to quickly determine the flotation properties of the individual components of a raw material. The Hallimond tube can also be used to study the mechanical entrainment of grains (Drzymala, 1994; Drzymala, 1994a; Drzymala, Lekki, 1989a and 1989b, Drzymala, 1999; Szyska et al., 2008; Wang et al., 2015; Konopacka, 2005).

The aim of this study was to investigate the flotation of selected minerals in terms of their natural hydrophobicity. Sulphur, silicon, fluorite and galena were tested for floatability in distilled water and in the presence of  $\alpha$  - terpineol ( $C_{10}H_{18}O$ ). Mineral flotation was performed for the 0.071- 0.125 mm fraction in the presence of distilled water and frother used at doses of 150 g/Mg and 300 g/Mg.

## 2. RESEARCH METHODOLOGY

The minerals used in the study were obtained from the Laboratory of Mineral Processing (LPK) of the Faculty of Geoengineering, Mining and Geology of Wrocław University of Technology (WGGG PWr), according to records with CAS numbers: 87 sulphur (S), CAS 241 silicon (Si), CAS 391 calcium fluorite - fluorite ( $CaF_2$ ) and CAS 29 lead sulphate - galena (PbS). According to literature, sulphur, silicon, fluorite and galena differ in hydrophobicity and thus in the contact angle (Figure 1).

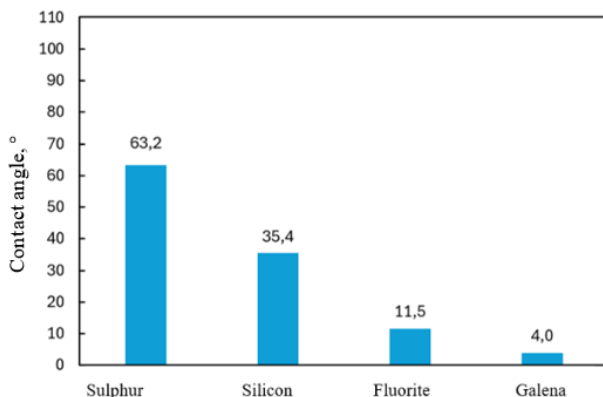


Figure 1. Natural hydrophobicity of minerals (Drzymala, 2009)

Sulphur is the most hydrophobic, with a contact angle of 63.2°. It is followed by silicon, also classed as highly hydrophobic, with a contact angle of 35.4°. The third

mineral in terms of hydrophobicity is fluorite. It is classified as a low-hydrophobic substance, as its contact angle varies between  $10^\circ$  and  $13^\circ$ . The least hydrophobic of the minerals studied is galena, which is classed as low-hydrophobic with a contact angle of only  $4^\circ$  (Drzymala, 2009).

The mineral samples were crushed in a laboratory mortar and then sieved using laboratory sieves to extract a grain class of 0.071- 0.125 mm, which formed the feed for the flotation (Figure 2).

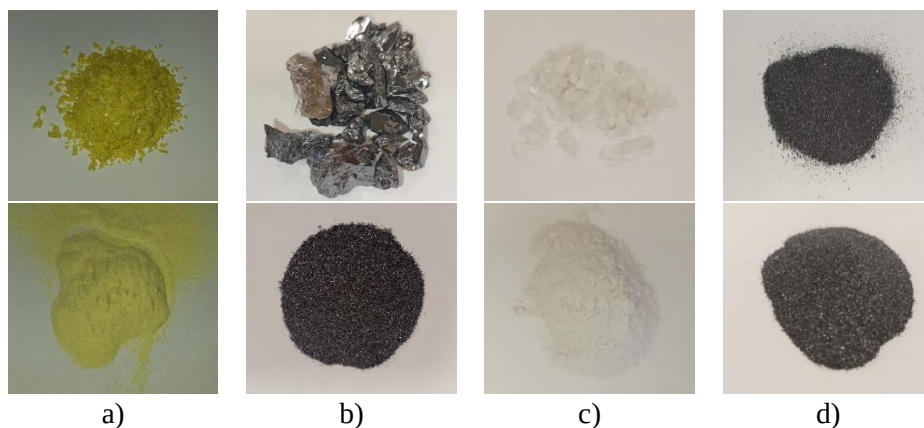


Figure 2. Mineral samples subjected to crushing and classification to prepare feed for flotation a) sulphur, b) silicon, c) fluorite, d) galena, (Błaszczów, 2025)

Flotation was carried out in a Hallimond tube with a cell height of 0.36 m, a cross-section of  $0.625 \cdot 10^{-3} \text{ m}^2$  and a volume of  $0.2 \cdot 10^{-3} \text{ m}^3$  with an air flow rate of 4.5- 4.6  $\text{dm}^3/\text{h}$ . Flotation tests were conducted in distilled water and in the presence of an aqueous solution of the frother  $\alpha$  - terpineol ( $\text{C}_{10}\text{H}_{18}\text{O}$ ) at a concentration of  $0.75 \text{ g}/\text{dm}^3$ . Each flotation was conducted for 35 minutes or until the entire sample had fully floated, meaning that 100% of it was recovered in the concentrate receiving section. During each flotation, the cumulative yield was analysed.

### 3. RESULTS DEVELOPMENT AND DISCUSSION

#### 3.1. FLOTATION OF MINERALS IN WATER AND IN AQUEOUS FROTHER SOLUTION

Figure 3 shows the flotation kinetics of the tested minerals in distilled water and in the presence of an aqueous frother solution. In all cases studied, an improvement in mineral flotation was observed with an added frother dose. Sulphur floated best and fastest, while galena floated worst.

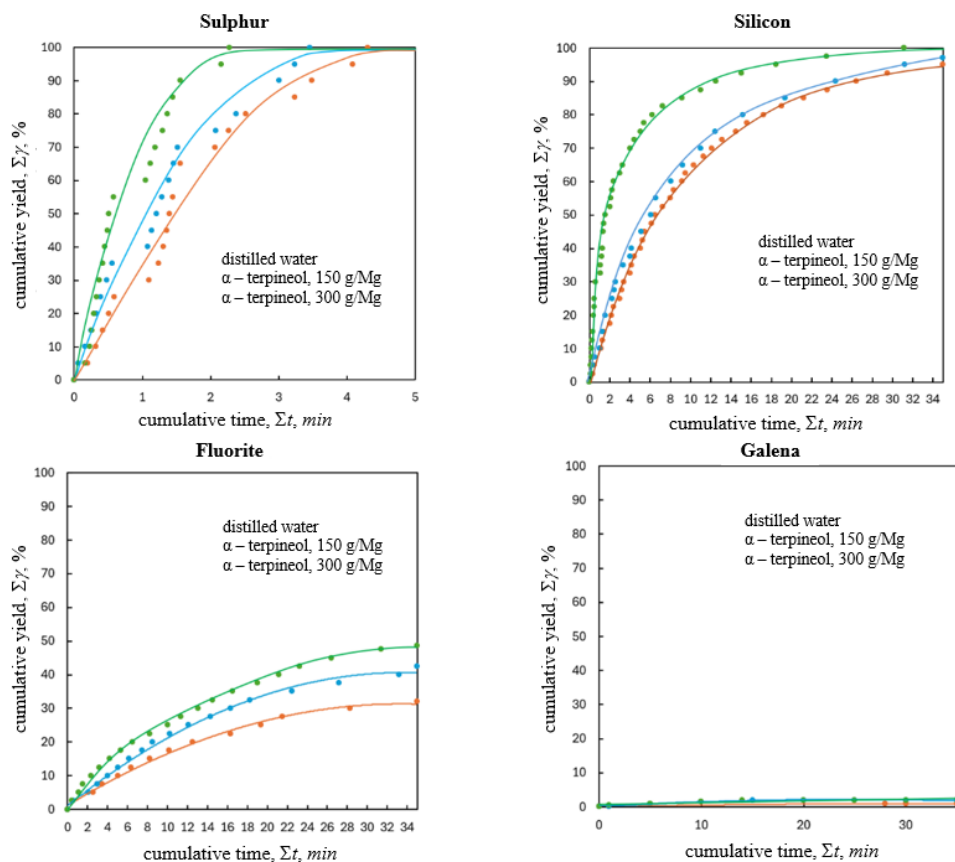


Figure 3. Flotation kinetics of a) sulphur, b) silicon, c) fluorite, d) galena in distilled water and in the presence of  $\alpha$ -terpineol frother at different concentrations

Figure 4 shows the correlation between the maximum yield of minerals such as sulphur (A), silicon (B), fluorite (C) and galena (D) and their flotation time. The analysis also considers the impact of the addition of  $\alpha$ -terpineol at 300 g/Mg on the analysed process. In the case of sulphur, both in its absence and in the presence of alpha-terpineol, a maximum yield of 100% was achieved in a very short time: in distilled water (continuous line) it took 4 minutes and 31 seconds, while with the frother (dashed line) at a dose of 300 g/Mg it took only 2 minutes and 28 seconds. The use of  $\alpha$ -terpineol at a concentration of 300 g/Mg reduced the sulphur flotation time by half. Silicon flotation showed improvement after the addition of alpha-terpineol. Without the frother, a maximum silicon yield of 95% was achieved after 35 minutes, while with  $\alpha$ -terpineol the time was reduced to 31 minutes and 15 seconds and the maximum yield increased to 100%. This shows that the addition of the frother has a positive, albeit small, effect on

the speed and efficiency of the silicon flotation process. In the case of fluorite flotation without the addition of  $\alpha$ -terpineol, the maximum yield was 32% after 35 minutes. The addition of the frother improved the fluorite flotation process, also increasing the maximum fluorite yield to 48.5%. In the case of galena flotation without as well as with the addition of alpha-thermineol at a concentration of 300 g/Mg, the maximum yield was very low and did not exceed 2%. With flotation in distilled water only, the yield reached a maximum value of 1%, while with the added frother it was 2%. Neither time nor the addition of a frother improved galena flotation. Among the four minerals analysed, a clear trend was observed for three of them: sulphur, silicon and fluorite, indicating that the presence of  $\alpha$ -terpineol improved the efficiency and acceleration of the flotation process.

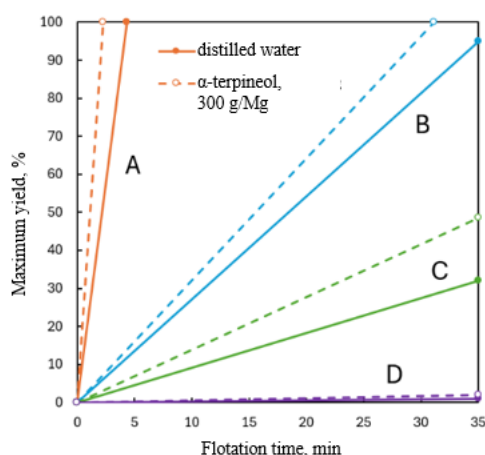


Figure 4. Dependence of the maximum yield of A) sulphur, B) silicon, C) fluorite, D) galena on the time of flotation in distilled water and with a 300g/Mg frother

### 3.2. DEPENDENCE OF MAXIMUM FLOTATION YIELD ON MINERAL DENSITY

Figure 5 shows the dependence of the maximum flotation yield of the studied minerals on their density. Sulphur, with a density of  $2.0 \text{ g/cm}^3$ , reached a maximum yield of 100%. This means that the flotation process was effective and very efficient. Whereas silicon, with a slightly higher density of  $2.3 \text{ g/cm}^3$ , also achieved a high yield of 95%. This indicates that silicon flotation was also effective and not much different from sulphur flotation. Fluorite, with a density of  $3.1 \text{ g/cm}^3$ , had a lower maximum yield of 32%. Galena, with a density of  $7.4 \text{ g/cm}^3$ , reached a maximum yield of only 1%, indicating that the galena flotation process was inefficient. Analysis of the graph shows that the efficiency of the flotation process decreased as the density of the substance increased. The higher the density of naturally hydrophobic minerals, the lower the efficiency of the flotation process.

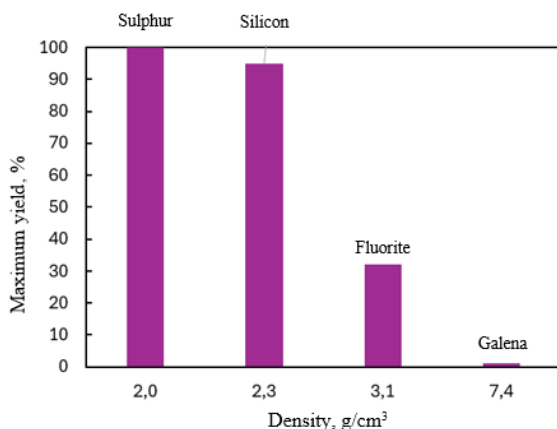


Figure. 5. Dependence of maximum flotation yield in distilled water on mineral density

### 3.3. IMPACT OF MINERAL HYDROPHOBICITY AND FROTHER DOSE ON THEIR MAXIMUM FLOTATION YIELD

Figure 5 shows the maximum flotation yield of sulphur, silicon, fluorite and galena as a function of their contact angle and  $\alpha$ -terpineol dose. Sulphur, which stands out with the biggest contact angle of  $63.2^\circ$  (Drzymała, 2009), reached a maximum yield of 100%, regardless of the alpha-terpineol dose applied. This indicates an efficient flotation process, which, based on the diagram, does not require a frother. Silicon, with a contact angle of  $35.4^\circ$ , reached a maximum yield of 95% in distilled water. In the presence of  $\alpha$ -terpineol at a concentration of 150 g/Mg, this yield increased to only 97%, and at a concentration of 300 g/Mg it reached 100%. The results indicate that the addition of  $\alpha$ -terpineol had a positive effect on the silicon flotation efficiency, however, the differences are small. Fluorite, with a contact angle, according to Drzymała (2009), of approximately  $11.0^\circ$ , had lower maximum flotation yield. In the case of distilled water, it reached only 32%. However, the introduction of alpha-terpineol raised the result to 40.5% at a concentration of 150 g/Mg and to 48.5% at a concentration of 300 g/Mg. It follows that fluorite flotation requires a frother to increase the efficiency of the process. Galena, with a contact angle around  $4.0^\circ$  (Drzymała, 2009), achieved the lowest maximum flotation yield. In distilled water, it reached only 1%. While in the presence of alpha-terpineol, at both the 150 g/Mg and 300 g/Mg doses, the maximum yield increased slightly to 2%. This shows that efficient flotation of galena under these conditions is not possible.

The flotation efficiency of the substance decreased as the contact angle decreased. Sulphur and silicon, characterised by bigger contact angles, floated easily, even without the use of chemical reagents. In the case of fluorite, the use of a frother was a useful

method to improve its flotation efficiency. Galena, on the other hand, was the least susceptible to the flotation process and the use of  $\alpha$ -terpineol produced low yield irrespective of the frothing reagent dose used.

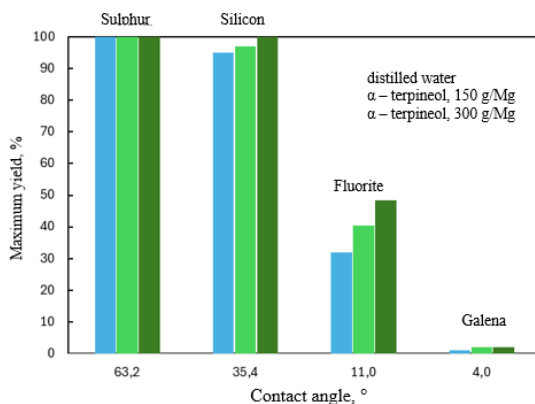


Figure 6. Dependence of maximum flotation yield on mineral hydrophobicity

#### 4. CONCLUSIONS

In this study, the flotation of naturally hydrophobic minerals such as sulphur, silicon, fluorite and galena was analysed in clear water and in the presence of a frother alpha-terpineol in a Hallimond tube.

The results showed significant differences in the floatability of the minerals studied. Sulphur, characterised by the highest hydrophobicity, achieved 100% yield in the shortest time. When a frother dose of 300 g/Mg was added, the maximum yield was obtained after almost 2.5 minutes of flotation.

Silicon, also classed as a highly hydrophobic mineral, achieved a high maximum yield of 100%; however, it required a longer flotation time. At a frother dose of 300 g/Mg, maximum yield was achieved after approximately 31 minutes.

Fluorite, characterised by low hydrophobicity, showed less susceptibility to flotation. In distilled water, it obtained a maximum yield of 32%, while the use of the frother at doses of 150 g/Mg and 300 g/Mg increased the yield to 40.5% and 48.5% respectively. The flotation process of fluorite was slower and less efficient compared to sulphur and silicon.

The least floatability was shown by galena, which is a low-hydrophobic mineral. Regardless of the flotation medium used (distilled water, frother 150 g/Mg and 300 g/Mg  $\alpha$ -terpineol), the maximum galena recovery did not exceed 2%. This result indicates very low susceptibility of galena to flotation under natural conditions and limited effectiveness of the frother used.

Analysis of the results also showed that flotation efficiency decreases with increasing mineral density (Figure.6.). Sulphur and silicon, with relatively low densities (2.0 g/cm<sup>3</sup> and 2.3 g/cm<sup>3</sup> respectively), achieved higher maximum yield compared to fluorite (3.1 g/cm<sup>3</sup>) and galena (7.4 g/cm<sup>3</sup>). Thus, it can be concluded that both natural hydrophobicity and mineral density are key factors influencing floatability.

The use of  $\alpha$  - terpineol as a frother significantly improved flotation efficiency for sulphur, silicon and fluorite. Higher frother doses contributed to a faster and more efficient flotation process, especially for sulphur and silicon. However, galena proved extremely difficult to float even with a frother.

The study concluded that:

1. The effectiveness of flotation depends on the natural hydrophobicity of the mineral substances. The greater the contact angle, the more effective and efficient the flotation process.
2. Flotation efficiency depends on the density of the minerals. The higher the mineral density, the less efficient the flotation process.
3. The addition of a frother improves flotation. A higher dose of  $\alpha$  - terpineol results in an accelerated and more efficient flotation of minerals, with the exception of galena.
4. Flotation time has an impact on its efficiency. Increasing the flotation time of minerals allows their flotation potential to be fully realised.

#### ACKNOWLEDGEMENTS

The author thanks Aleksandra Błaszaków for participation in measurements.

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