Sulfur Enrichment in Biodiesel Precipitates: Possible Causes and Impacts

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Biodiesel is widely used as an additive to fossil diesel because it is a renewable and biodegradable biofuel that reduces pollutant emissions. Although considered a clean fuel, concerns persist regarding its use due to its tendency to form precipitates that can damage automotive components. Therefore, like any other fuel, biodiesel must meet a series of specifications to ensure its quality for commercialization. One such specification is the maximum sulfur content, as this element is a significant contributor to the greenhouse effect, as well as to corrosion in equipment and damage to automotive engines. Thus, this study aimed to analyze the sulfur concentration in biodiesel samples using the ICP-OES technique. The results showed that the precipitate samples had a higher sulfur content than their respective supernatant samples.
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Since 2008, the addition of biodiesel to fossil diesel has been mandatory in Brazil, as it is a renewable and non-toxic biofuel that also provides greater lubricity to diesel. Biodiesel (B100) can be obtained through the transesterification reaction between a triglyceride (which may be of animal or vegetable origin) and a primary alcohol (such as methanol or ethanol), producing glycerol as a byproduct [1].

However, despite its advantages, biodiesel still presents some challenges due to its composition, such as its ability to absorb moisture and its higher tendency to form precipitates. The formation of precipitates in biodiesel not only compromises its quality and that of diesel-biodiesel blends (BX) but can also cause clogging of automotive filters and injector valves [2].

Despite these challenges, B100 remains one of the most produced biofuels in Brazil [3] due to its low aromatic content, high cetane number, and low sulfur content, among other characteristics that make it an eco-friendly fuel [4]. Nevertheless, to maintain these advantages, it is important to monitor its properties to ensure its quality continuously. Maintaining fuel quality prevents deposit formation, increases equipment durability, and reduces pollutant emissions.

In Brazil, fuel quality is regulated by the National Agency of Petroleum, Natural Gas, and Biofuels (ANP), which ensures adherence to performance and safety standards. One of the ANP's specifications for biodiesel is the maximum sulfur content, which must not exceed 10 mg kg^{-1} [5]. If not monitored, high sulfur levels can cause corrosion in pipelines and storage units, damage vehicle engines, and have environmental impacts associated with the emission of sulfur oxides (SO_x) [6].

This work aimed to analyze the sulfur concentration in the supernatant and precipitate samples of biodiesel and investigate its possible causes using an inductively coupled plasma optical emission spectrometer ICP-OES.

Materials and Methods

After collecting 500 mL of biodiesel sample, the aliquot was transferred to a distillation funnel and left to stand for seven days to allow the precipitate to separate. Both the precipitate and the supernatant were then collected and stored in separate Falcon tubes. This procedure was carried out for eight biodiesel samples, designated as BD-X, where BD represents "biodiesel" and X represents the sample number. The sulfur content

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was measured using an inductively ICP-OES iCAP PRO XP Duo, Thermo Scientific.

Results and Discussion

The concentrations of sulfur content in the biodiesel samples are shown in Figure 1. We observe an increase in sulfur content from the supernatant to the precipitate of the same biodiesel sample. One possible explanation is that sulfur originates from contaminants in the raw material in the form of a compound with low solubility in biodiesel, which may precipitate over time, thereby enriching the biodiesel precipitate with sulfur [7].

Another possible explanation is the presence of microbial activity. Although sulfur can act as an inhibitor of microbial action in diesel and diesel/biodiesel blends, sulfate-reducing bacteria (SRB) may metabolize sulfur and form insoluble compounds such as iron sulfide (FeS). These bacteria can use sulfate (SO_4^{2-}) to generate sulfide ions (S^{2-}) in solution, which precipitate instantly [8].

Additionally, sulfur may react with other metals such as iron, copper, and zinc to form metallic sulfides, which are known for their very low solubility [9].

Although studies have shown that high sulfur concentrations can inhibit microbial growth in fossil diesel and biodiesel mixtures due to their inhibitory properties [6], there are still few studies in the literature addressing the significance of sulfur concentration in pure biodiesel. Even though biodiesel is virtually sulfur-free, the element can be extracted along with vegetable oils in the form of phospholipids and glucosinolates [7].

Conclusion

In all samples, the sulfur content exceeded the maximum limit established by ANP and was even higher in the precipitate samples compared to their corresponding supernatant samples. This increase may be related to the concentration of sulfur in impurities originating from the raw material and/or the biodiesel production process, the presence of microbial colonies, or possible reactions of sulfur with other metals resulting in the formation of metallic sulfides.

Further research is needed to confirm the origin of sulfur and identify the cause of the increased sulfur content in the precipitate compared to the remaining biodiesel liquid.

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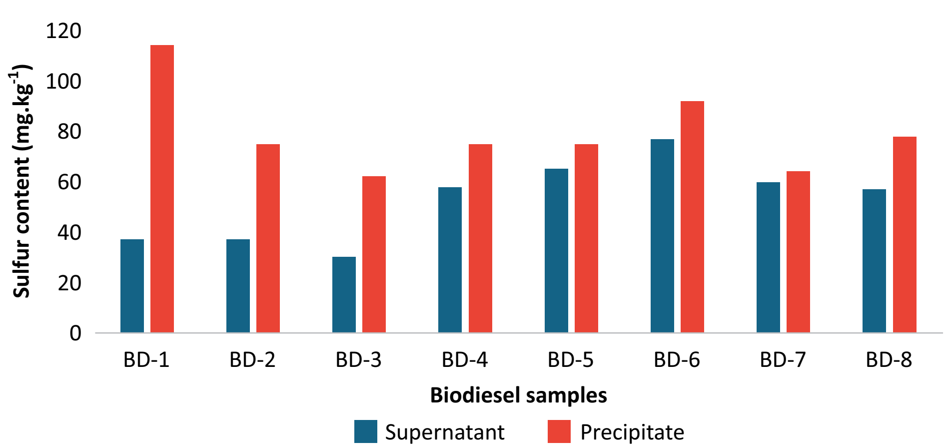
Figure 1. Sulfur content by biodiesel sample (mg kg^{-1}).



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