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Influence of Reactor Scale on the Properties of Sulfurized Olive Pomace Oil Additives

Roland NAGY
University of Pannonia
Gábor Zoltán NAGY
University of Pannonia
Ditta Adrienn Gerbovits
gerbovits.ditta.adrienn@mk.uni-pannon.hu

University of Pannonia

Short Report

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Abstract

Sulfurized vegetable oils are widely used as ashless extreme-pressure (EP) lubricant additives. This study examined the scale-up synthesis of sulfurized olive pomace oil (OPO) in geometrically similar Büchi reactors at 50 g, 500 g, and 5000 g scales. OPO was reacted with 10 wt% sulfur and 1 wt% dodecyl amine at 140–150°C. Scale-up increased kinematic viscosity at 40°C from 288.8 to 1484 mm²/s, while active sulfur content decreased from 4.20 to 2.12 wt%. Nevertheless, all additives showed excellent EP performance, with weld loads of 2200–2300 N in four-ball tests at 3 wt% treat rate in SN 150 oil. Olive pomace oil is therefore a promising sustainable feedstock for EP additive production.

Statement of Novelty

Although sulfurized vegetable oils are established lubricant additives, much less is known about how their production changes when laboratory synthesis is scaled up. This work addresses that practical gap using olive pomace oil, an agro-industrial byproduct, as a renewable feedstock. By increasing batch size from 50 g to 5 kg in geometrically similar reactors, we show that reactor scale strongly affects viscosity and the amount of reactive sulfur, while high load-carrying performance is preserved. The study therefore demonstrates that effective extreme-pressure additives can be produced from olive pomace oil beyond small laboratory batches and identifies viscosity control and sulfur reactivity as key issues for future pilot-scale and industrial process development.

1. Introduction

Sulfurized vegetable oils are biodegradable ashless extreme-pressure (EP) additives capable of forming protective metal-sulfide tribofilms under severe lubrication conditions [1–3]. Olive pomace oil (OPO), a byproduct of olive oil production, represents a promising sustainable feedstock because of its high oleic acid content and suitable unsaturation for sulfurization reactions [4,5].

Although laboratory-scale sulfurization processes are well established, scale-up may significantly influence heat transfer, mixing efficiency, and viscosity development during synthesis [6]. The present work investigates the influence of reactor scale on the physicochemical and tribological properties of sulfurized OPO additives.

2. Experimental

Sulfurization reactions were performed in geometrically similar Büchi glass reactors at 50 g, 500 g, and 5000 g batch scales. Olive pomace oil was reacted with 10 wt% elemental sulfur using 1 wt% dodecyl amine catalyst. Reactions were carried out at 140–150°C for 1–3 h under a maximum pressure of 2 bar.

The synthesized additives were characterized using standard analytical methods:

- kinematic viscosity: EN ISO 3104,

- total sulfur content: ASTM D5185,
- active sulfur content: ASTM D1662,
- four-ball weld load: DIN 51350-4.

Tribological performance was evaluated in SN 150 base oil containing 3 wt% additive concentration.

3. Results and Discussion

The physicochemical and tribological properties of the synthesized additives are summarized in Table 1.

Table 1
Effect of batch scale on sulfurized olive pomace oil additives

Batch size (g)	KV40 (mm²/s)	Total sulfur (wt%)	Active sulfur (wt%)	Weld load (N)
50	288.8	8.88	4.20	2300
500	432.2	8.14	2.40	2200
5000	1484	8.93	2.12	2200

The viscosity increased substantially with increasing reactor scale, particularly between the 500 g and 5000 g batches. This behavior is likely associated with reduced mixing efficiency and localized sulfur overreaction during larger-scale synthesis.

In contrast, total sulfur incorporation remained relatively constant across all scales. However, active sulfur content decreased significantly during scale-up, indicating possible changes in sulfur bonding structures and reduced concentrations of reactive sulfur species.

Despite these physicochemical differences, all additives maintained high extreme-pressure performance. Four-ball weld load values remained above 2200 N at only 3 wt% treat rate in SN 150 base oil, indicating preserved load-carrying capability.

Figure 1.: *Influence of reactor scale on kinematic viscosity at 40°C and active sulfur content of sulfurized olive pomace oil additives.*

The results demonstrate that sulfurized olive pomace oil synthesis can be successfully scaled up; however, viscosity management becomes increasingly important at pilot-scale production.

4. Conclusions

The present study demonstrates the feasibility of scaling up sulfurized olive pomace oil synthesis from laboratory to pilot scale. Reactor scale significantly influenced viscosity and active sulfur content, while total sulfur incorporation and tribological performance remained relatively stable. The synthesized

additives exhibited high load-carrying capacity in lubricating oil formulations, confirming the suitability of olive pomace oil as a sustainable feedstock for sulfurized EP additive production.

Declarations

Conflict of Interest

The authors declare that they have no competing financial or non-financial interests that could have influenced the work reported in this paper.

Author Contribution

R.N. and G.Z.N. performed the experimental work and contributed to data analysis. D.A.G. contributed to the study design, data interpretation, and preparation of the manuscript. All authors reviewed and approved the final manuscript.

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Availability of Data and Materials

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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Figures

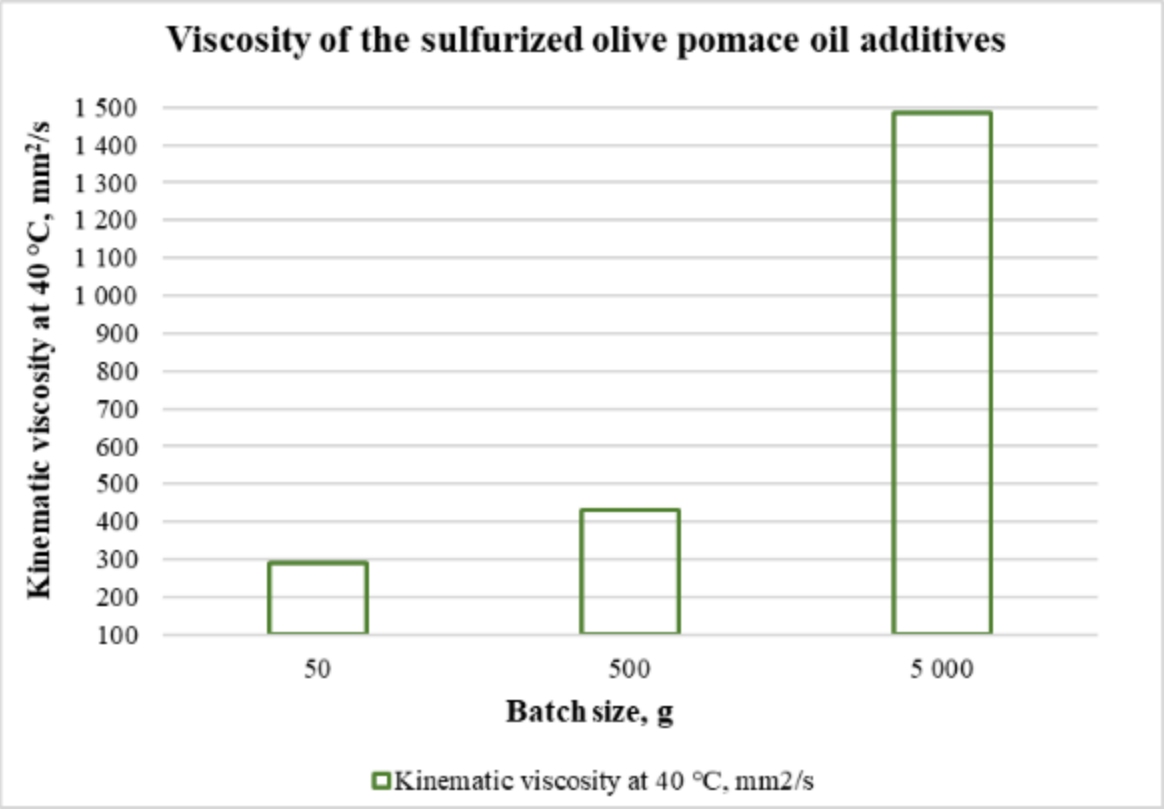


Figure 1

Influence of reactor scale on kinematic viscosity at 40 °C and active sulfur content of sulfurized olive pomace oil additives