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Analytical Investigation of Oleic Acid Double Bond Cleavage Using Cu-$BiVO_4$ Photocatalyst in Water/Ethanol System Using GC-MS Analysis

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Abstract

The oxidative cleavage of unsaturated fatty acids is a promising pathway for producing value-added chemicals; however, its photocatalytic conversion remains limited by catalyst efficiency. This study investigates the performance of $BiVO_4$ and Cu-modified $BiVO_4$ (Cu–$BiVO_4$) in promoting the photocatalytic cleavage of oleic acid in a water–ethanol system under visible light irradiation. The catalysts were synthesized via hydrothermal and impregnation methods, followed by photocatalytic reactions for 24 h. Product distribution and transformation pathways were analyzed using GC–MS. The results show that pristine $BiVO_4$ exhibits limited catalytic activity, while Cu–$BiVO_4$ significantly enhances the formation of C14 and C16 saturated fatty acids along with various oxidative cleavage products. This improvement is attributed to enhanced charge separation and increased generation of reactive oxygen species. These findings demonstrate that Cu–$BiVO_4$ is a more effective photocatalyst for the oxidative transformation of unsaturated fatty acids.

Keywords: $BiVO_4$-phtocatalyst, fatty acid, GC-MS analysis, Oleic acid oxidation, Photocatalytic cleavage.

Introduction

Bismuth vanadate ($BiVO_4$) is a promising semiconductor oxide-based photocatalyst material for the degradation of organic compounds under visible light, thanks to its narrow band gap (~2.4 eV), good chemical stability, and its ability to generate reactive radicals such as •OH and $•O_2^-$ (Kamble et al., 2023; Sajid et al., 2018). Its monoclinic crystal structure is known to be more effective in facilitating charge separation than the tetragonal phase. Monoclinic $BiVO_4$ with an olive-like morphology has been reported to exhibit high photocatalytic activity in the degradation of organic compounds under visible light irradiation (Zhu et al., 2016). Understanding these structural properties has encouraged the development of various modification strategies for $BiVO_4$ to improve its performance in oxidative reactions.

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One of the widely developed approaches is the modification of $BiVO_4$ with co-catalysts, to overcome the main limitation of rapid recombination of electron–hole pairs. Gomes et al. (2023) showed that the addition of unsupported $Co(OH)_2$ nanoparticles to the surface of $BiVO_4$ was able to significantly increase the photocatalytic efficiency in organic oxidation reactions (Gomes et al., 2022). Meanwhile, Onwudiwe et al. (2024) developed a $Ce_2O_3/BiVO_4$ composite embedded in graphene oxide (rGO), and successfully increased the photocatalytic activity in the degradation of complex organic pollutants (Onwudiwe et al., 2021). Structural modification and the incorporation of co-catalysts have proven to be key strategies to improve the performance of $BiVO_4$, including in the cleavage of chemical bonds such as C–C or C=C in complex organic molecules(Cui et al., 2021; Lih et al., 2025; Zhang et al., 2023).

In addition to the degradation of organic pollutants, $BiVO_4$ has also begun to be explored in biomass transformation based on carbon bond cleavage reactions. Liu et al. (2024) reported that $BiVO_4$ modified through surface fluoridation was able to initiate the cleavage of glycerol C–C bonds to form formic acid through a photoelectrochemical reaction (Liu et al., 2024). Furthermore, Li et al. (2025) showed that nanoporous $BiVO_4$ on a photoanode electrode promoted the cleavage and reformation of C–C bonds during glycerol oxidation, confirming the potential of this material in facilitating complex radical transformations (Hilbrands et al., 2023). These findings expand the potential applications of $BiVO_4$ not only in the degradation of simple molecules, but also in the processing of biomolecular substrates such as unsaturated fatty acids.

Oleic acid (C18:1) is an unsaturated fatty acid that has one cis double bond (Garti & Avni, 1982; Li et al., 2018), making it an ideal substrate for the study of photocatalytic double bond cleavage reactions (Enferadi Kerenkan et al., 2016; Soutelo-Maria et al., 2018a, 2018b; Spannring et al., 2014). Practically, oleic acid is known to be easily soluble in ethanol – as reported in the esterification study by Alkahlawy et al. (2025), which showed that the oleate–ethanol reaction takes place in a homogeneous phase (Alkahlawy & Gaffer, 2025). This solubility is very advantageous in $BiVO_4$-based photocatalytic studies because it allows effective interaction of substrate molecules with the catalyst surface without the need for additional surfactants. In addition, the use of ethanol as a solvent is highly compatible with GC–MS-based analysis, which can detect and identify volatile products resulting from the fragmentation and cleavage of oleic acid double bonds during oxidative reactions (Hewavitharana et al., 2020). Thus, the $BiVO_4$ system in ethanol medium offers a powerful platform for exploring advanced photocatalytic reactions, particularly in the controlled transformation of lipid molecules.

Despite extensive studies on $BiVO_4$-based photocatalysts for organic degradation and biomass transformation, their application in the selective oxidative cleavage of long-chain unsaturated fatty acids such as oleic acid remains underexplored. In particular, the role of Cu as a co-catalyst in enhancing C=C bond cleavage efficiency and directing product selectivity has not been systematically investigated. Therefore, this study aims to evaluate the photocatalytic performance of Cu–$BiVO_4$ in oleic acid oxidation and to elucidate the resulting product distribution using GC–MS analysis.

Method

$BiVO_4$ Photocatalyst Synthesis.

$BiVO_4$ photocatalyst synthesis was carried out using a hydrothermal procedure adapted from a protocol validated by (X. Jin et al., 2018). The material was prepared as described in our previous work. In brief, equimolar amounts (3 mmol) of $Bi(NO_3)_3.5H_2O$ and NH_4VO_3 were dissolved in 2 M HNO_3 with the pH adjusted to 1.5–2.0. The mixture was reacted at 200 °C for 24 h in a Teflon-lined autoclave. The formation of the monoclinic scheelite phase, which is crucial for photocatalytic activity, was confirmed in the study.

Surface Modification.

$BiVO_4$ surface modification was carried out using an impregnation method with Cu as the dopant, following the approach reported by (Xu et al., 2008). In a typical procedure, 2.5 mmol $BiVO_4$ was mixed with 0.125 mmol of $M(NO_3)_2 \cdot xH_2O$ (M = Co, Ni, Cu), corresponding to a molar ratio of $M/BiVO_4$ = 5%. The mixture was dispersed in 10 mL of distilled water and stirred at approximately 65

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°C to ensure homogeneous distribution of the metal precursor on the $BiVO_4$ surface. The resulting suspension was then dried and subsequently calcined in a furnace at 300 °C for 4 h to obtain the modified photocatalyst.

Oleic Acid Double Bond Cleavage Oxidation.

The photocatalytic test reaction for the cleavage of the oleic acid double bond (C=C) was carried out using the methodology reported by (Gomes et al., 2022) and (Han et al., 2019). A total of 0.05 g of photocatalyst (0.15 mmol) was dispersed in two solvent systems, a water/ethanol mixture, to evaluate the effect of polarity on substrate adsorption dynamics and oxidative activity. 1 mL of oleic acid was added to the suspension, and the mixture was incubated in the dark for 1 hour to reach adsorption–desorption equilibrium on the particle surface. Next, air was supplied as an oxidizing agent, and the suspension was stirred at 500 rpm to ensure homogeneity of particle distribution and light exposure. Irradiation was performed using an 18 W Philips TLD lamp with a spectral range of 380–680 nm for 24 hours.

Product Analysis by GCMS.

The reaction products were analyzed using an Agilent 7820A GC–MS instrument operated with a multistage heating program to maximize the separation of volatile and semi-volatile compounds from the reaction. Component separation procedure refers to (Hajra et al., 2017). The performed using a step-by-step oven temperature program, starting at 60°C for 3 minutes, then increasing the temperature to 200°C at a rate of 20°C/minute and holding for 2 minutes. The temperature was then increased again to 230°C at a rate of 15°C/minute and held for 10 minutes.

The reaction products were analyzed using an Agilent 7820A GC–MS instrument operated with a multistage heating program to maximize the separation of volatile and semi-volatile compounds. Component separation procedure followed the protocol by Hajra et al. (2017). To ensure the validity of the investigation, the sensitivity of the analysis was evaluated by determining the Limit of Detection (LOD) and Limit of Quantitation (LOQ) based on the signal-to-noise (S/N) ratio, processed using OriginPro software. The LOD was established at an S/N ratio of 3:1, and the LOQ was established at an S/N ratio of 10:1. Only peaks with an S/N ratio exceeding the LOQ threshold were used for relative area normalization and selectivity calculations. The oven temperature program started at 60 °C for 3 minutes, increased to 200 °C at 20 °C/minute (held for 2 minutes), and finally reached 230 °C at 15 °C/minute (held for 10 minutes).

Results and Discussion

$BiVO_4$ synthesized via hydrothermal methods is widely reported to form a monoclinic scheelite phase, which is known to exhibit high photocatalytic activity. Furthermore, surface modification with Cu was carried out to improve the efficiency of electron-hole pair separation and expand its reactivity (Chen et al., 2016; Zhao et al., 2025). The photocatalytic activity was then evaluated through the double bond cleavage reaction in oleic acid. The reaction products were analyzed using GC–MS to determine compositional changes, fragmentation (Wang et al., 2025), and the formation of new compounds as indicators of photocatalytic effectiveness (Lestari & Maulida, 2024; Phuruangrat et al., 2023; Prasetiyo et al., 2025).

GC-MS analysis of standard oleic acid and photocatalytic reaction products can be seen in **Figure 1,** showing significant changes in the chromatographic profile when $BiVO_4$ and $BiVO_4$–Cu catalysts were used. The chromatogram of standard oleic acid displayed a dominant peak at RT ≈ 19.6 min, confirming the purity of the starting material. Under reaction conditions using the catalyst, the oleic acid peak decreased, as seen in **Figure 2**. The $BiVO_4$ catalyst chromatogram was still dominated by the residual oleic acid peak at RT ≈ 19.4 min, but a new peak appeared at RT > 30 min, indicating that the conversion was still limited and the photocatalytic activity of $BiVO_4$ was not efficient enough in promoting the cleavage of the C=C double bond. The use of $BiVO_4$–Cu resulted in a much more striking change, indicating enhanced photocatalytic activity compared to pristine $BiVO_4$. This is a strong indicator of effective double bond cleavage. This behavior is consistent with previous studies

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reporting that Cu acts as an electron trap, improving charge separation and enhancing reactive oxygen species generation. At RT ≈ 13.9, a peak resulting from double bond cleavage was identified, producing C14, and at RT ≈ 16.5, a peak from C16. This is a strong indicator of effective double bond cleavage. Furthermore, the emergence of several new peaks in the RT range of 20–40 minutes—which were not observed in the standard sample or the pure $BiVO_4$ catalyst—reflects the formation of compounds resulting from oxidative cleavage.

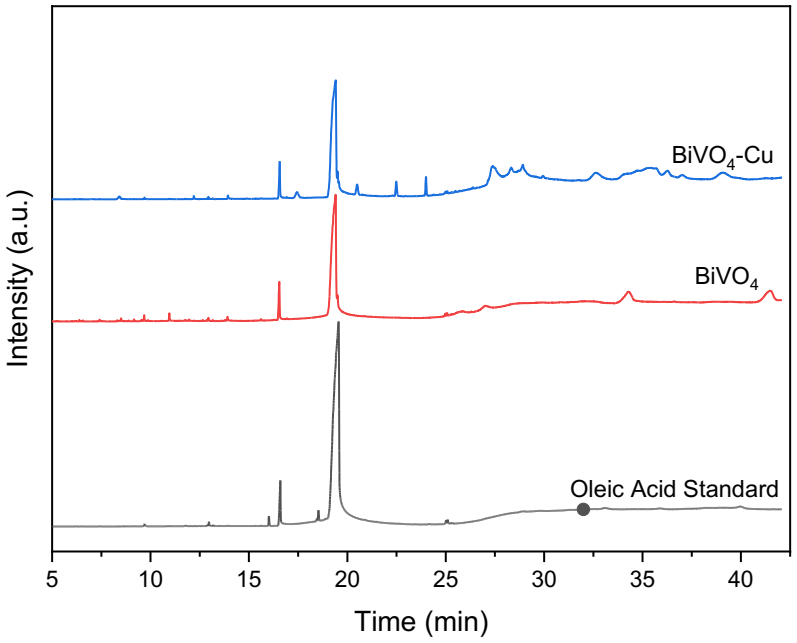

Figure 1. Oxidation chromatogram of double bond cleavage of oleic acid

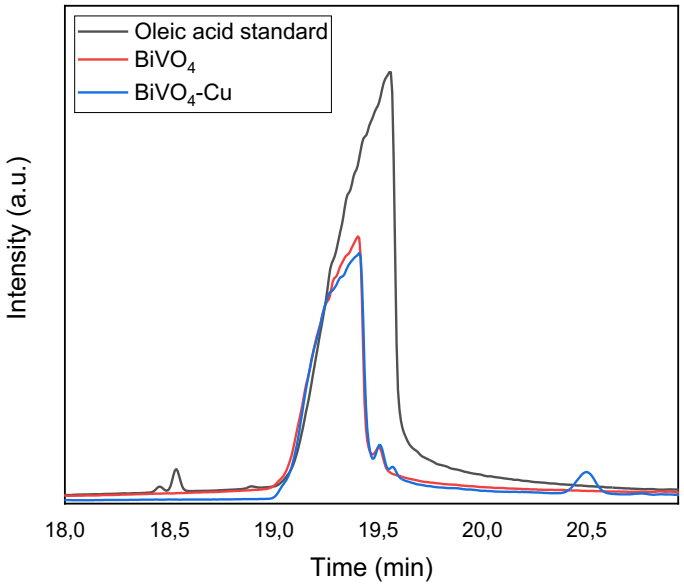

Figure 2. Chromatogram of oleic acid peak

The fragmentation pattern observed in oleate oxidation products indicates a tendency for double bond cleavage through attack by reactive oxygen species at the allylic position, a common pathway in the oxidation of unsaturated fatty acids. This mechanism is consistent with the fundamental

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understanding that peroxyl radicals formed during autoxidation can trigger rearrangement and β-scission, resulting in aldehydes, ketones, or carboxylic acids as end products (Gardner, 1989). Meanwhile, vanadium-based catalysts are known to be able to activate oxygen and facilitate oxidative scission in olefins through oxygen transfer and the formation of radical and peroxometalate intermediates, which lead to the formation of short-chain carbonyl compounds (Upadhyay et al., 2021; Yun et al., 2021a). This finding is in line with the characteristics of $BiVO_4$ as an n-type photocatalyst that is capable of producing high-energy holes (h^+) and superoxide radicals ($\bullet O_2^-$) through the reduction of O_2, where both species play a role in the oxidation and cleavage of C–C bonds in organic substrates (Hilbrands et al., 2023; L. Jin et al., 2023). The presence of alcohol-based solvents such as ethanol has been reported to stabilize peroxide-type intermediates and enhance the selectivity toward aldehydes and other carbonyl compounds during C=C bond cleavage. This behavior is consistent with the DFT study (Xiao et al., 2023), which demonstrated that the oxidation of oleic acid proceeds through the formation of hydroperoxide (ROOH), peroxyl (ROO•), and alkoxyl (RO•) radicals, and that polar media such as ethanol can stabilize these intermediates, thereby promoting fragmentation toward carbonyl-containing products. Likewise, (Yun et al., 2021b) showed that oxidative cleavage of olefins catalyzed by metal–peroxide systems yields aldehydes and carboxylic acids, supporting the notion that peroxide intermediates play a central mechanistic role in the overall oxidative scission process. These findings collectively suggest that alcohol solvents may facilitate radical-driven pathways by stabilizing key oxygenated intermediates. The consistency between the product patterns obtained in this study and those reports reinforces that the dominant mechanism is the oxidative cleavage of double bonds via a radical pathway based on active oxygen species generated on the $BiVO_4$ surface.

GC–MS analysis of the reaction products over the $BiVO_4$–Cu catalyst is presented in **Table 1**. The results were evaluated based on relative peak area normalization. Oleic acid remains the dominant component, indicating that conversion could not be quantitatively determined under the current analysis conditions. However, several reaction products were detected, including oxidation products (25.44%), chain-breaking products (25.56%), and heavy compounds (30.34%), suggesting that multiple reaction pathways, such as oxidation, cracking, and polymerization, occurred during the reaction.

Table 1. Product distribution and selectivity from GC–MS analysis over $BiVO_4$–Cu catalyst.

RT (min)	Compound	Area Relatif (%)	Category	Selectivity (%)
13.9	Tetradecanoic acid (C14)	0.68	Minor	0.85
16.5	n-Hexadecanoic acid (C16)	7.34	Fatty acid	9.19
19.4	Oleic acid	100	Unreacted	—
27.3	Oxidation products	20.33	Oxidation	25.44
28.9	C14-C16 fatty acid	6.89	Fatty acid	8.62
34.8	Chain breaking products	20.43	Cracking	25.56
35.4	Heavy compounds (C>18)	24.25	Polymerization	30.34

The selectivity of each product was calculated based on the normalized relative peak area, excluding the unreacted oleic acid, according to Equation 1. This approach allows for a comparative evaluation of product distribution, assuming that the GC–MS peak area is proportional to the concentration of each compound.

$$Selectivity\ (\%) = \left(\frac{area\ of\ a\ given\ product}{total\ area\ of\ all\ detected\ products}\right) \times 100\% \quad (1)$$

Products in the C14–C16 carbon range detected at RT 28.9 min (6.89%) indicate the occurrence of double bond cleavage and partial hydrogenation, leading to the formation of medium-chain fatty acids. Furthermore, chain-breaking products with a selectivity of 25.56% (RT 34.8 min) suggest that the catalyst effectively promotes C–C bond cleavage, generating smaller molecular fragments from oleic acid. In addition, the formation of heavy compounds (C>18) with a selectivity of 30.34% (RT 35.4 min) indicates the occurrence of secondary reactions, such as polymerization or condensation, resulting in higher molecular weight products.

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Based on **Table 2**, the GC–MS results in this study indicate that the Cu co-catalyst in $BiVO_4$ significantly increases the photocatalytic activity, so that the photocatalytic oxidative cleavage reaction takes place more effectively and produces a more complex product distribution compared to $BiVO_4$. The presence of Cu as a cofactor in $BiVO_4$ increases the photocatalytic reactivity, accelerating the cleavage of the C=C double bond in oleic acid into saturated fatty acids and shorter chains (C14–C16). However, most of the oleic acid remains, indicating a partial reaction.

Table 2. Comparison of GCMS data with and without catalyst

Parameter	Oleic Acid Standard	$BiVO_4$	Cu-BiVO4
Main Peak	19.55 min – Oleic Acid	19.39 min – Oleic Acid	19.39 min – Oleic Acid
New product (short chain)	-	-	C14, C16 fatty acids
Peak count > 30 min	-	some (oxidation)	Many (chain-breaking products)
Efficiency of double bond cleavage	-	less efficient	less efficient

Conclusion

This study demonstrates that Cu modification significantly enhances the photocatalytic performance of $BiVO_4$ for oleic acid oxidative cleavage. The Cu–$BiVO_4$ catalyst promotes efficient C=C bond scission, resulting in the formation of medium-chain fatty acids (C14–C16) and diverse oxidation products. The improved activity is attributed to enhanced charge separation and increased generation of reactive oxygen species. These findings highlight the potential of Cu–$BiVO_4$ as an effective photocatalyst for biomass-derived fatty acid valorization.

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