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Dear Professor Yuhang Li,

Thank you for the submission of your revised manuscript. As in other cases like this, we have sent the revised manuscript back to the original reviewers. In light of their positive re-assessment, we are delighted to inform you that your manuscript 8227073, titled "Electrosynthesis of Glycine from Nitrate and Glyoxylic Acid over a Bi₂S₃ Nanosheets-based Catalyst", has been accepted for publication in *Chemistry - A European Journal*.

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Reviewer comments:

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Reviewer 1 Comments to the Author

The authors have addressed all the comments and thus the ms can be accepted as it is.

Reviewer 2 Comments to the Author

The authors have carefully revised the manuscript and have fully addressed all my previous concerns. The revised version is significantly improved. I recommend acceptance for publication

RESEARCH ARTICLE

Electrosynthesis of Glycine From Nitrate and Glyoxylic Acid Over a Bi₂S₃ Nanosheets-Based CatalystXulin Nong¹ | Chunqi Yang¹ | Jingwen Zhuang¹ | Yuhang Li^{1,2} ¹Key Laboratory for Ultrafine Materials of Ministry of Education, School of Materials Science and Engineering, East China University of Science and Technology, Shanghai, China | ²Department of Chemical Engineering, School of Chemistry and Chemical Engineering, Shanghai Jiao Tong University, Shanghai, China

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ABSTRACT

The green and efficient synthesis of amino acids is of great importance to both life science and the chemical industry. Electrocatalytic C–N coupling, driven by renewable electricity under mild conditions, offers a sustainable route for converting simple feedstocks into value-added nitrogen-containing compounds and thus provides a promising strategy for glycine production. Bismuth-based catalysts are commonly used in the electrosynthesis of amino acids. Herein, we report a Bi₂S₃ nanosheet-based catalyst for glycine electrosynthesis under acidic conditions, achieving 67.5% Faradaic efficiency (FE) and a yield rate of 0.51 mmol h^{−1} cm^{−2} at 200 mA cm^{−2}. Operando Raman and attenuated total reflectance surface-enhanced infrared absorption spectroscopy (ATR-SEIRAS) identify catalyst reconstruction and a tandem mechanism mediated by NH₂OH and oxime intermediates. Combined with the control sulfurization experiment on Bi, we found that sulfur incorporation can modulate the product selectivity, possibly by influencing the subsequent hydrogenation of the oxime intermediate to glycine.

1 | Introduction

Amino acids play important roles in both biological systems and diverse industrial applications, and they have therefore attracted broad interest for their potential in green and efficient synthetic routes [1, 2]. The electrocatalytic formation of C–N bond provides an attractive method for sustainable amino acid synthesis, since it allows simple substrates to be converted into high-value nitrogenous compounds with renewable electricity under gentle conditions [3–6]. Among these approaches, electrocatalytically coupling nitrate with glyoxylic acid to produce glycine is notably promising due to its environmental friendliness, as it provides an alternative to the Strecker synthesis, which relies on toxic cyanide [7, 8]. However, this process involves multiple complex steps, including the multi-step reduction of nitrate, C–N coupling, and subsequent hydrogenation, leading to low selectivity for the target product and slow reaction kinetics [9–12]. Therefore, designing an electrocatalyst that can efficiently promote nitrate reduction and

precisely guide the subsequent C–N coupling and hydrogenation steps is a key challenge for advancing this technology.

Bismuth (Bi) based materials have shown considerable promise for nitrate reduction relative to other non-noble metal catalysts, owing mainly to their unique electronic properties, high hydrogen evolution reaction (HER) overpotential, and excellent stability [13–15]. Notably, Bi-based catalysts are conducive to the selective formation of hydroxylamine intermediates, which play a critical role in subsequent C–N coupling [16–19]. Nevertheless, efficient glycine electrosynthesis still requires precise regulation of intermediate adsorption and conversion, especially in the downstream hydrogenation step. In this context, the incorporation of sulfur into Bi can effectively modulate its catalytic properties. The combination of low electronegativity, relatively large atomic radius, and strong polarizability enables sulfur to tune the electronic structure associated with the Bi active sites [20–22]. Thus, the adsorption and transformation of key