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RECYCLED IRON SLUDGE FROM HOMOGENEOUS FENTON TREATMENT AS A LOW-COST CATALYST FOR WATER TREATMENT APPLICATIONS

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ABSTRACT

The increasing application of Fenton-based advanced oxidation processes for treating high-strength industrial effluents, such as bioethanol wastewater, generates substantial amounts of iron sludge as a secondary byproduct. Although commonly regarded as waste, this iron-rich material offers potential for reuse as a catalytic precursor within a circular economy framework. This study investigates the physicochemical properties of iron sludge derived from homogeneous Fenton treatment of bioethanol wastewater and evaluates its feasibility for catalytic reuse. The sludge was characterised using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM). FTIR analysis revealed Fe–O bonds, surface hydroxyl groups, and residual organic functionalities, indicating the presence of chemically active sites. XRD results confirmed γ-FeOOH (lepidocrocite) as the dominant phase, accompanied by partially crystalline iron-based structures associated with redox activity. SEM images showed irregular, aggregated particles with porous and rough surface morphologies favourable for heterogeneous catalytic reactions. To assess practical applicability, the recovered iron sludge was reused in a Fenton-like process, achieving 84.72% COD removal, compared to 97.63% obtained using fresh catalyst in a conventional homogeneous Fenton system. These findings demonstrate that iron sludge generated from Fenton treatment can be effectively transformed into a low-cost, functional iron-based catalyst. The study provides important insights into the sustainable management of Fenton-derived waste, supports the development of waste-derived catalytic materials, and contributes to advancing environmentally responsible water treatment technologies.

Keywords: Circular Economy, Iron-based Catalyst, Fenton Process, Waste Valorisation, Wastewater Treatment.

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INTRODUCTION

The bioethanol industry has grown substantially in recent years, driven by the rising demand for renewable energy sources and initiatives to reduce greenhouse gas emissions. In 2023, global ethanol production reached approximately 116 billion litres, with the United States and Brazil accounting for 80% of this output. India emerged as the third-largest producer, tripling its output since 2018 to 6.4 billion litres.[1] The International Energy Agency (IEA) projects that biofuel demand will expand by 38 billion litres over the 2023-2028 period, a near 30% increase from the previous five years. Emerging economies like Brazil, Indonesia, and India are expected to lead this growth due to robust biofuel policies, rising transport fuel demand, and abundant feedstock potentials such as corn and sugarcane.[2] However, the byproduct of the bioethanol production process poses a significant environmental hazard if not adequately treated. One major environmental byproduct of bioethanol production is vinasse, or the distillery

wastewater, a high-strength effluent known for its elevated organic load, low biodegradability, and complex chemical composition. With an estimated production rate of 10 to 14 litres of bioethanol wastewater per litre of ethanol generated, improper discharge can lead to serious issues, including soil and water contamination due to its high concentrations of organic matter and nutrients.[3,4] Such effluents can exhibit organic loads reaching 27,500 to 299,250 mg chemical oxygen demand/L under high-temperature conditions, further amplifying their polluting potential.[5] Traditional methods of treating bioethanol wastewater, such as storage in lagoons, have been criticised for generating greenhouse gas emissions and negatively impacting soil and aquifer structures during fertirrigation practices.[6] In the context of bioethanol wastewater treatment, several methodologies have been developed to manage its high organic load and associated environmental risks. While traditional methods such as biological treatments, including anaerobic digestion and various physicochemical processes, have been widely used, these methods exhibit significant limitations. One of the main drawbacks of anaerobic digestion is its slow adaptation of microbial populations to bioethanol wastewater conditions. This adaptation requires extended periods, which can delay the efficiency of organic matter removal and biogas production.[7] Additionally, anaerobic processes may not fully mineralise recalcitrant compounds often found in the wastewater, leading to incomplete treatment and potential toxicity issues in the resulting effluent.[8] Physicochemical treatments, including the use of oxidants such as ozone, are limited by their operational complexity.[9] Among the various treatment methods, advanced oxidation processes (AOPs) homogeneous Fenton treatment, are increasingly employed to treat bioethanol wastewater due to their effectiveness in degrading refractory organic compounds. The Fenton reaction employs hydrogen peroxide (H_2O_2) in combination with ferrous ions (Fe^{2+}) to produce hydroxyl radicals (•OH) that effectively oxidise organic pollutants, including those found in bioethanol wastewater.[10,11] The formation of hydroxyl radicals during this process is pivotal in breaking down persistent contaminants, including phenolic compounds that hinder the use of bioethanol wastewater in microbial cultures.[12] This makes the Fenton process a practical choice for treating bioethanol wastewater, as it not only reduces the concentration of recalcitrant organic matter but also improves the biodegradability of the resulting effluent, facilitating subsequent biological treatments such as anaerobic digestion.[13] However, these processes result in the formation of iron sludge as a secondary waste stream, which is typically discarded despite its rich iron content. Iron sludge recovered from Fenton systems can serve as a valuable resource rather than a waste product. From a circular economy perspective, this iron sludge presents a promising opportunity for resource valorisation, particularly as a potential low-cost catalyst for further oxidation processes. One practical opportunity is to recycle the iron in the sludge back into the Fenton reaction itself. This not only mitigates disposal issues but also reduces operational costs[14], as the expenditure on iron reagents is minimised.[15] The recycling of iron sludge generated during the homogeneous Fenton treatment has gained significant attention due to some benefits, such as tackling waste management issues associated with Fenton sludge and allowing for the reuse of iron as a catalyst in wastewater treatment processes. Another strategy includes the transformation of Fenton sludge into iron-based composite materials or catalysts. For instance, studies have reported the successful transformation of iron sludge into magnetically separable catalysts, which can facilitate easier recovery and reuse in heterogeneous Fenton processes.[16] Such materials not only allow the integration of the Fenton process into wastewater treatment systems more seamlessly but also provide enhanced reaction kinetics due to the increased surface areas[17] and available active sites.[18] Nevertheless, the successful transition of such sludge into a viable catalyst material requires a thorough understanding of its physicochemical properties, including surface morphology, crystallinity, and functional groups. These properties provide information about its catalytic behaviour in advanced oxidation systems. This study aims to comprehensively characterize iron sludge obtained from the Fenton treatment of bioethanol wastewater using a combination of FTIR, XRD, and SEM. These analytical techniques provide insights into the microstructure of the sludge, phase composition, and chemical bonding environment. This paper aims to assess the potential of the material for reuse as a heterogeneous or homogeneous catalyst and to establish a foundation for future application studies. By studying the structural and chemical attributes of this waste-derived material, this research contributes to the development of sustainable pathways for industrial byproduct utilisation in environmental remediation

technologies. This research also aligns with the United Nations Sustainable Development Goals by supporting SDG 6 (Clean Water and Sanitation) through improved wastewater treatment performance, SDG 12 (Responsible Consumption and Production) via the valorization of iron sludge within a circular economy framework, and SDG 9 (Industry, Innovation, and Infrastructure) through the development of innovative, low-cost catalytic materials for environmental applications.

EXPERIMENTAL

Material and Methods

The materials used include iron sludge was collected as a byproduct of a homogeneous Fenton oxidation process conducted on real bioethanol wastewater obtained from a local distillery in Yogyakarta, Indonesia. A 500 mL beaker glass was used as the reactor, placed on a magnetic stirrer and operated at a constant stirring speed of 200 rpm to ensure proper mixing as depicted in Fig.-1. The initial pH of wastewater was adjusted to approximately 3.0 using H_2SO_4 (Merck) to optimise hydroxyl radical generation. Subsequently, the Fenton reaction was performed by adding ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$, Merck) as the iron source, followed by the addition of hydrogen peroxide (H_2O_2, 30% w/w, PT PIP Indonesia) to initiate the reaction. The molar ratio of $[H_2O_2]$: $[Fe^{2+}]$ and the dosages were determined based on preliminary optimisation studies.[19]

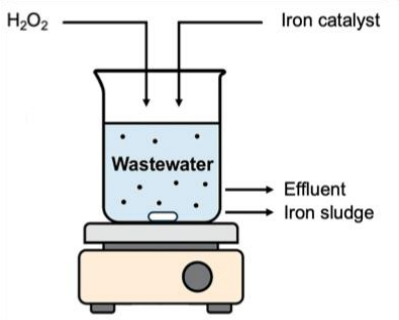

Fig.-1: Experimental Apparatus Layout of Fenton Treatment of Bioethanol Wastewater

The reaction was allowed to proceed for 60 minutes under constant stirring at room temperature. After the reaction time had elapsed, the system was neutralised to pH 7.0 by the gradual addition of 1 M NaOH solution. This pH adjustment assisted in quenching the Fenton reaction and promoting the precipitation of ferric hydroxide species, resulting in the formation of iron sludge. The precipitated iron sludge was then allowed to settle for 24 hours, separated by vacuum filtration, and washed thoroughly with deionised water to remove residual soluble impurities and organic matter. The sludge was then oven-dried at 105°C for 12 hours and stored in a desiccator until further analysis.

Iron Sludge Characterisation

Chemical bonding and functional groups present in the iron sludge were identified using Fourier Transform Infrared Spectroscopy equipped with an attenuated total reflectance (FTIR-ATR) setup (Shimadzu IRXross, Japan). Crystalline phase identification of the iron sludge was performed using an X-ray diffractometer (XRD) (Rigaku MiniFlex 600, Japan). Surface morphology and microstructural features of the dried iron sludge were observed using Scanning Electron Microscopy (SEM) (Hitachi SU3500, Japan).

Catalyst Reuse in Fenton Reaction

To assess the catalytic performance of the iron sludge, a comparative degradation experiment was conducted using the dried sludge as a recycled catalyst in a Fenton-like reaction and compared with a conventional homogeneous Fenton system using fresh $FeSO_4 \cdot 7H_2O$. Both systems were tested under identical operating conditions, including pH 3.0, constant H_2O_2 and iron catalyst dosage, and a reaction time of 60 minutes. In the sludge-based system, a defined mass of the recycled catalyst was added directly into raw bioethanol wastewater samples along with H_2O_2. After treatment, the change in COD removal efficiency was measured to evaluate the treatment performance using different catalysts for the

assessment of the practical reuse potential of Fenton-derived iron sludge as a low-cost, sustainable catalyst for wastewater treatment.

RESULTS AND DISCUSSION

Functional Group Analysis by FTIR

Figure-2 shows the FTIR spectrum of the iron sludge generated from homogeneous Fenton treatment of bioethanol wastewater, which shows distinct vibrational bands that confirm the presence of both inorganic and organic functional groups. This spectrum provides valuable information about the functional groups and the implications for catalytic reuse. The broad bands observed in the 3200–3500 cm^{-1} range are assigned to O–H stretching vibrations, which reflect the presence of hydroxyl groups and adsorbed water.[20] These hydroxyl groups are commonly associated with hydrated ferric oxides and play a critical role in catalytic reactions, as they can act as active sites for the adsorption and activation of hydrogen peroxide in the Fenton-like process. Additional peaks near 1400–1450 cm^{-1} are indicative of carboxylate groups ($–COO^-$), which suggest the adsorption of intermediate or residual organic compounds from the bioethanol wastewater. Meanwhile, a significant peak around 1650 cm^{-1} may be attributed to O–H bending or C=C stretching, suggesting the presence of moisture and residual aromatic compounds.[21] These surface groups can improve the catalyst dispersion in aqueous media and facilitate better contact between active sites and reactants. Furthermore, a noticeable peak observed around 590 cm^{-1} corresponds to Fe–O stretching vibrations, indicating the presence of iron oxide and hydroxide structures.[22] These iron-containing phases are essential for maintaining redox cycling between Fe^{2+}/Fe^{3+} during catalytic applications in the Fenton system. The ability of these materials to participate in electron transfer reactions is important for the generation of hydroxyl radicals (•OH) in subsequent reuse scenarios. These findings on the FTIR analysis reveal that the sludge contains functional groups of surface hydroxyls, Fe–O bonds, and adsorbed organic compounds, which can be beneficial to redox reactions, further supporting its applicability as a catalyst.

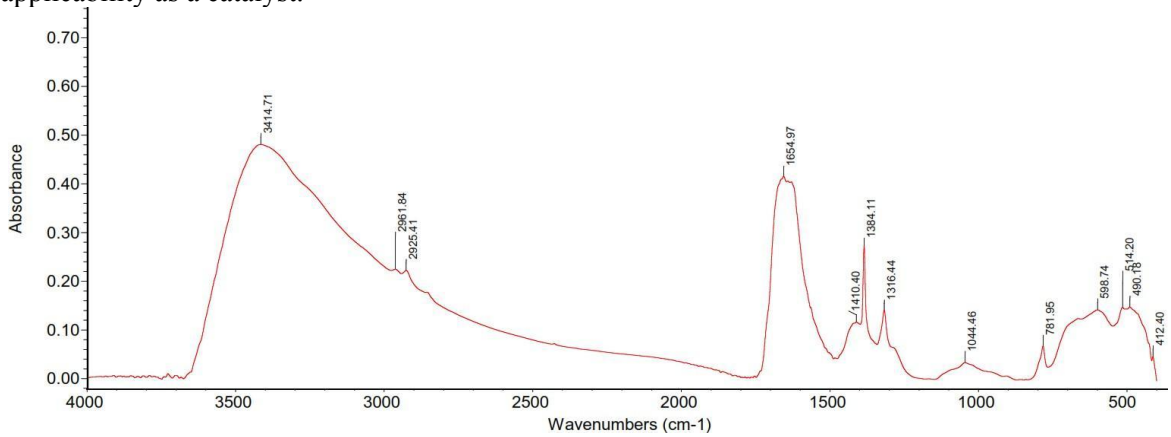

Fig.-2: FTIR Spectrum of the Iron Sludge Derived from Fenton Treatment of Bioethanol Wastewater

Crystalline Phase Identification by XRD

The crystalline structure of the iron sludge was evaluated using XRD, and the diffractogram is presented in Fig.-3, which reveals a mixture of crystalline and amorphous phases formed during the Fenton reaction. The major diffraction peaks correspond to the formation of γ-FeOOH (lepidocrocite). Lepidocrocite is a common iron oxyhydroxide phase produced through the oxidation of Fe^{2+} and hydrolysis of Fe^{3+} under acidic to neutral pH conditions in Fenton reactions.[23,24] Additional peaks with partial overlap suggest the presence of an amorphous iron oxide phase, and possibly mixed ferrite-type structures resembling $(Pr_{0.8}Sr_{0.2})FeO_3$. Although present in smaller quantities, this material is known for its high oxygen mobility and electron conductivity, which are advantageous in advanced oxidation reactions. The moderate background noise in the pattern also indicates partial amorphous content, possibly due to residual organic matter or poorly crystallised phases. The presence of these phases in the XRD results indicates that the sludge may possess favourable electronic and catalytic properties, supporting its potential reuse as an iron-based catalyst and suitability for recycling as a Fenton-like catalyst.

Morphological Characterisation by SEM

The surface morphology of the fresh catalyst or $FeSO_4 \cdot 7H_2O$ (before Fenton treatment) and the iron sludge (after Fenton treatment) was examined using SEM, as shown in Fig.-4. Figure-4(a) represents the surface before Fenton treatment, which shows relatively compact and smooth layers with dense-plate structures and limited porosity.

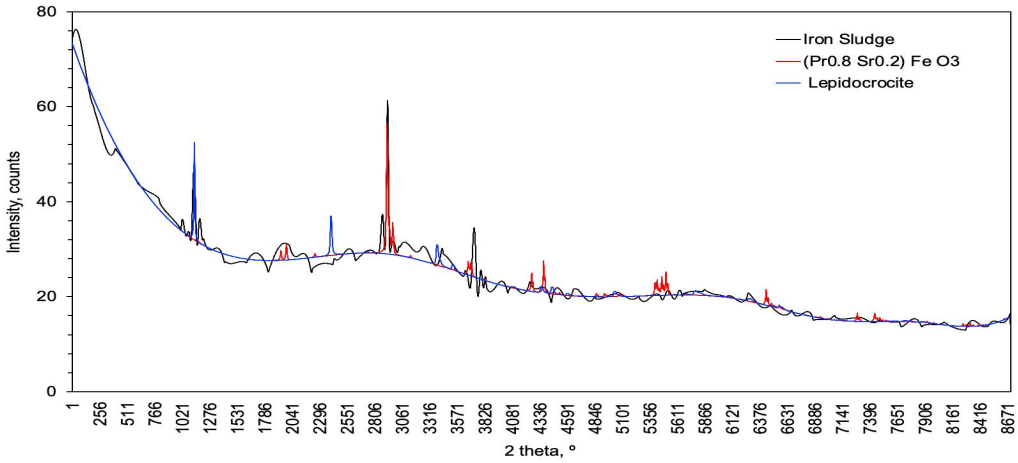

Fig.-3: XRD Pattern of the Iron Sludge

The surface appears to be structurally intact with minimal roughness, suggesting that the material was in a stable state with low surface activity. In contrast, Fig.-4(b) shows the iron sludge formed after the Fenton reaction, which reveals a significantly altered surface. The morphology of post-treatment catalyst shows major fragmentation, with the appearance of angular, irregular particles and well-developed surface roughness. The small and granular deposits and cracks on the surface indicate the formation of precipitated ferric hydroxides. These features correspond to the hydrolysis and polymerisation of Fe^{3+} ions as the pH was adjusted to neutral (~7), leading to the formation of insoluble iron precipitates. Such morphology is also characteristic of iron oxides and hydroxides precipitated under acidic oxidative conditions, where rapid nucleation and co-precipitation processes occur.[3,25] Moreover, the surface profile of iron sludge shows the irregularly shaped particles with coarse, porous surfaces and significant agglomeration. The substantial shift from a smooth to a rough and fractured morphology not only confirms the chemical transformation of iron species during Fenton oxidation but also highlights the physical changes induced by the radical reaction and subsequent precipitation. These surface modifications are important indicators of this iron sludge for catalytic reuse.[26] The increased surface roughness and porosity in this material suggest a relatively high surface area, which may enhance its adsorptive and catalytic capabilities if reused in a Fenton-like process in future applications.

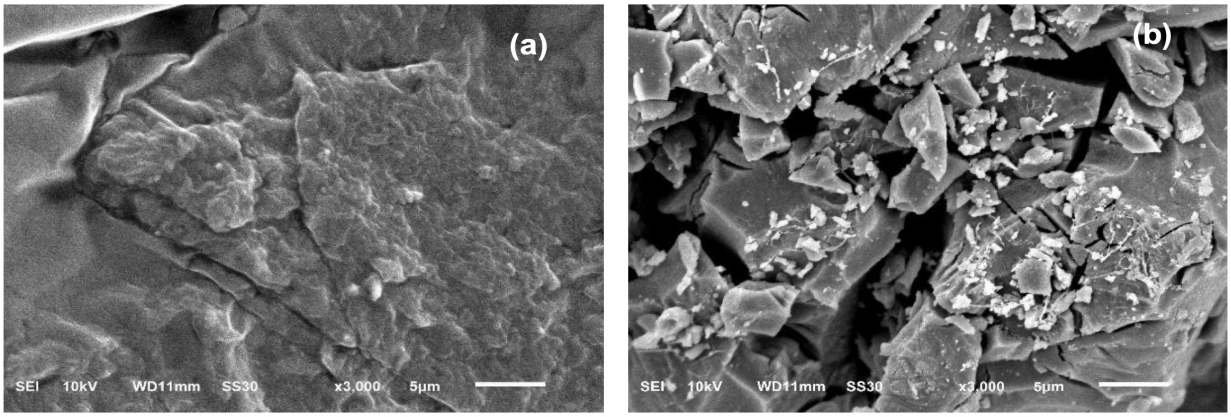

Fig.-4: SEM Micrograph of: (a) Fresh Catalyst and (b) Iron Sludge Obtained from Fenton-Treated Bioethanol Wastewater at 3000× Magnification

Evaluation of Catalytic Performance of Recycled Iron Sludge

To validate the applicability of the Fenton-derived iron sludge, it was reused as a catalyst in a subsequent Fenton-like reaction. The treatment performance was compared to a control system employing a conventional homogeneous Fenton process using fresh $FeSO_4 \cdot 7H_2O$. The results are presented in Table-1. The recycled catalyst system achieved a COD removal efficiency of 84.72%, which was comparable to the fresh homogeneous system, 97.63%. While slightly lower in absolute removal, the reused sludge demonstrated remarkable catalytic activity, indicating that the active Fe species in the sludge (e.g., Fe^{2+}/Fe^{3+} and γ-FeOOH) remained functional after initial use. Although the fresh catalyst showed higher degradation rates, the recycled catalyst exhibited sustained oxidative performance, which can be attributed to the redox activity of the iron oxyhydroxide phases confirmed by XRD, and the reactive surface sites identified by FTIR. The porous morphology observed in SEM analysis likely supported hydrogen peroxide activation via surface-mediated mechanisms. These findings demonstrate that recycling iron sludge not only reduces secondary waste but also maintains effective catalytic performance, thereby offering a sustainable alternative to fresh iron dosing in Fenton-based treatments. This supports the feasibility of integrating iron sludge reuse into circular wastewater management systems.

Table-1: Comparison of COD Removal Efficiency between the Fresh and Recycled Iron Sludge as the Catalyst after Homogeneous Fenton Treatment

System	COD Wastewater		
	Initial (mg/L)	Final (mg/L)	Removal (%)
Fresh catalyst ($FeSO_4.7H_2O$)	71495	1690	97.63
Recycled iron sludge	71495	10920	84.72

The results obtained in this study demonstrate clear relevance to the Sustainable Development Goals. The high COD removal efficiency achieved using recycled iron sludge in a Fenton-like process (84.72%) directly supports SDG 6 (Clean Water and Sanitation) by demonstrating effective treatment of high-strength industrial wastewater. The successful reuse of iron sludge as an iron-based catalyst highlights progress toward SDG 12 (Responsible Consumption and Production) through waste valorisation and reduction of secondary waste generation. Furthermore, the identification of catalytically active iron phases and favourable surface morphology from an industrial byproduct contributes to SDG 9 (Industry, Innovation, and Infrastructure) by enabling the development of low-cost, scalable, and resource-efficient treatment materials. These results collectively demonstrate the practical alignment of these research outcomes with global sustainability targets.

CONCLUSION

This study demonstrated the potential of iron sludge, a byproduct of the homogeneous Fenton process applied to bioethanol wastewater, to be repurposed as a low-cost catalyst. Comprehensive physicochemical characterisation using FTIR, XRD, and SEM revealed that the sludge consists of irregular, porous particles with crystalline phases such as γ-FeOOH (lepidocrocite) and $(Pr_{0.8}Sr_{0.2})$ FeO_3 structures. The presence of Fe–O bonds, hydroxyl groups, and residual organic functionalities indicated chemically active surface sites suitable for catalytic applications. The catalytic performance of the recycled iron sludge was further evaluated in a Fenton-like reaction and compared to a conventional homogeneous Fenton system using fresh Fe^{2+}. Although slightly lower in COD removal efficiency, the recycled catalyst demonstrated substantial oxidative capability and improved BOD/COD ratio, indicating enhanced biodegradability of the treated effluent. These findings support the feasibility of reusing iron sludge as a functional iron-based catalyst in wastewater treatment processes, aligning with circular economy and sustainable waste management principles. Future studies may further explore regeneration cycles, surface modification, and kinetic modelling to optimise its catalytic application.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation.* All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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