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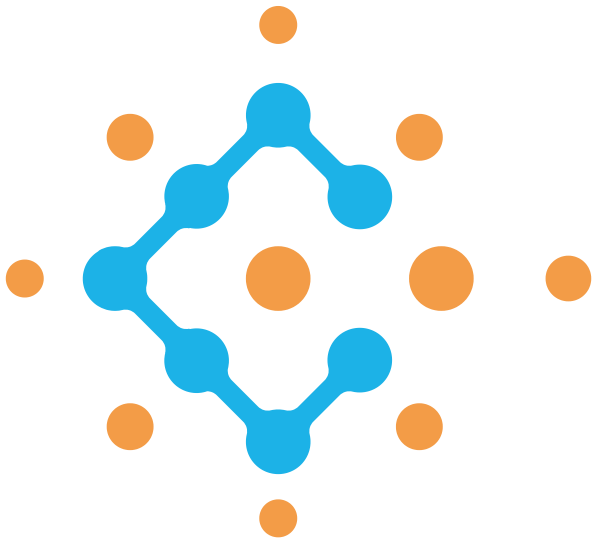
Featured Article

Enhanced Hydrogen Production using Zirconium Based MOFs.

p. 10



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Board Message

Dear Esteemed Readers,

It is with immense pride and excitement that I welcome you to the second issue of volume 2 of our society's technical journal. Building on the foundation laid in our inaugural issue, this edition signifies our unwavering commitment to fostering innovation, sharing knowledge, and strengthening connections within our professional community.

As our field continues to evolve, so too does the scope of ideas and challenges we face. This journal remains dedicated to providing a platform where groundbreaking research, practical insights, and diverse perspectives converge. It is our hope that these contributions spark dialogue, inspire action, and drive meaningful progress.

In this issue, you will find an enriched selection of articles that delve deeper into the complexities of our discipline. Each piece reflects the dedication and expertise of our contributors, as well as the collective vision of our society to lead and inspire. I encourage you to explore the content thoughtfully and consider how these insights might shape your work and broaden your understanding.

The success of this endeavor would not be possible without the support and collaboration of our authors, reviewers, and editorial team. Their hard work and passion have once again made this journal a testament to the vibrancy and depth of our community.
As we move forward, let us continue to celebrate the spirit of curiosity, innovation, and excellence that unites us. Thank you for your ongoing engagement and support. Together, we are shaping the future of our field.

Warm regards,

Letter From The Editor

Dear Colleagues,

Welcome to the second issue of Volume 2 of the Journal of Chemicals Research and Innovation Society. As we embark on another year of scientific exploration and knowledge sharing, we are excited to continue our mission of fostering groundbreaking research and collaboration in the chemical sciences.

Building on the foundation laid in our previous volumes, this issue presents a diverse collection of studies that showcase innovative methodologies, novel discoveries, and practical advancements across various fields of chemistry. Each contribution reflects the dedication and expertise of our research community, and we are proud to provide a platform for such impactful work.

I extend my deepest gratitude to our authors, reviewers, and editorial team for their unwavering commitment to excellence. Your contributions ensure that this journal remains a trusted source of scientific progress and a hub for meaningful discourse.

As we move forward, I encourage you to actively engage with the journal, whether through submitting your research, participating in discussions, or sharing insights with your peers. Together, we can continue to drive innovation and push the boundaries of chemical research.

Thank you for your continued support, and I look forward to the discoveries and advancements that lie ahead.

Best regards,

Shakeel Ahmed, Ph.D.,
Editor-in-Chief
Journal of Chemicals Research and Innovation Society

Developing and Validating Method for Online Monitoring of THPS-Based Biocides

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Abstarct

Aramco treats and transports millions of barrels of seawater over thousands of kilometers of pipeline network every day for reservoir injection. The high sulfate concentration in seawater can promote sulfate-reducing bacteria (SRB), which can lead to costly problems such as fouling, plugging, corrosion and reservoir souring. Biocide products such as glutaraldehyde- or THPS-based biocides are injected into the transportation pipeline for microbial control. To monitor the treatment effectiveness and optimize the treatment program, biocide residual concentrations at various downstream locations of the pipeline network were monitored via manual sampling and analysis using commercial test kits. However, sample collection, delivery, and analysis for biocide residual monitoring is a huge challenge for this large seawater injection system, especially for remote injection wells.

In this paper we will present and validate an innovative method based on permanganate-THPS reaction, which can be developed into an online analyzer for real-time monitoring of THPS-based biocides. The method was validated in a laboratory mock-up system with reaction chamber, flow cell, and the absorption spectrometer. The dynamic detection range in the mock-up system is approximately 20-1000 ppmv of THPS.

Introduction

It is a common practice in the Middle East region for the oil and gas companies to use seawater for reservoir injection for pressure maintenance. The typical Arabian Gulf seawater (AGS) has higher total dissolved solids (TDS) and sulfate than typical seawater, at approximately 45,000 mg/L TDS and 3,200 mg/L of sulfate [1]. The AGS at Qurayyah has an average TSD 57,700 mg/L and sulfate 4,280 mg/L, and millions of barrels of Qurayyah seawater is injected every day into Saudi Aramco oil reservoirs for pressure maintenance. The presence of high sulfate concentration can promote sulfate-reducing bacteria (SRB), which are considered one of the most damaging microbes in oil and gas industry [2-5]. Therefore, the seawater is treated with various chemicals and processes before it is injected into the reservoir. The typical treatments include disinfectant (e.g., sodium hypochlorite) to kill living organisms in the seawater, various physical and chemical processes (e.g., screens, flocculant, coagulant, filtrations) to remove solids and small particles, deaeration process (e.g., nitrogen, oxygen scavenger)

to remove the dissolved oxygen to the level less than 10 ppb. Finally, the organic biocides are injected into the transportation pipeline for microbial control in the treated seawater [6-7]. Tetrakis (hydroxymethyl) phosphonium sulfate (THPS) is a quaternary phosphonium compound with good environmental profile. It is degradable and non-bioaccumulative in the environment [8-10]. It is a broad-spectrum biocide, effective in pH range of 3-11, with a mode of action by reacting with specific amino acids on the cellular membrane of microorganisms. THPS is widely used as biocide in oil and gas industry for microbial control [11], and one of the most common active ingredients in the biocide products used in Aramco operations.

Saudi Aramco operates the world's largest seawater transportation pipeline network with various pipeline diameters, pressures, and flow rates. To monitor the treatment effectiveness and optimize the treatment program of THPS-based biocide, the samples have to be collected from various downstream locations of the pipeline network to determine the THPS biocide residual concentration and microbial counts such as SRB. However, sample collection, delivery, and analysis for biocide residual monitoring is a huge challenge for this large seawater injection system, especially for remote injection wells. More than often, the samples collected at the estimated biocide arrival time at the downstream locations showed no biocide residuals, one of the reasons being the difficulty to accurately estimate the arrival time due to the size and complexity of the pipeline network and flow fluctuation. An online analyzer which detects THPS biocide residuals automatically will provide a real time monitoring of residual concentration at downstream locations of a large pipeline network, eliminating the needs and challenges for manual sampling and analysis.

THPS-based biocides are traditionally detected and quantified using iodometric titration method with commercially available test kit [12]. In this paper we will present an innovative method, which can be developed into an online analyzer for real-time monitoring of THPS-based biocides.

Materials and Methods

Bis[tetrakis(hydroxymethyl)phosphonium] sulfate (THPS) solution (70-75% in H_2O) (Cat. No. 15175), potassium permanganate (Cat. No. 223468), and Hydrochloric acid solution (1.0 M) (Cat. No. H9892) were purchased from Sigma-Aldrich. Phosphate-buffered saline (PBS) (10X) (Cat. No. AM9625) was purchased from ThermoFisher. The biocide product (Duracide 9231) used in the experiments is supplied by one of the Saudi Aramco vendors. The product contains 20-40% THPS, 10-20% ethylene alcohol, and 10-20% proprietary non-surfactant-based component according to the product's Material Safety Data Sheet (MSDS). Milli-Q water was from Milli-Q IQ 7005 Pure & Ultrapure Water Purification System (Merck Corporation).

Design Principle:

THPS is a reducing agent. It reacts with potassium permanganate ($KMnO_4$, a strong oxidizer) in a manner that would decolorize the purple permanganate solution as a function of the THPS concentration. The mole ratio of the reaction between THPS and permanganate was approximately 2:1, meaning that 2 mole THPS would be able to decolorize 1 mole of KMnO4.

The color change can be measured by optical absorption at the maximum wavelength 525 nm. By measuring the absorption change of the permanganate solution after the reaction, the THPS concentration in the biocide products, and hence the biocide residual concentration in the water system, can be determined.

Validation of Permanganate-THPS Reaction

THPS was prepared at 0 to 1000 ppmv in Milli-Q water and Qurayyah AGS. 1.0 mM of acidified permanganate solution ($KMnO_4$·HCl, pH 5.0) was prepared in demineralized water, and added with 8.3 ml of 1 M hydrochloric acid (HCl) solution. Equal volumes (1.5 mL) of the THPS-containing samples and permanganate solution were mixed and allowed to react for 2 minutes at room temperature. Then, the intensity of the permanganate absorption at 525 nm was measured and normalized by subtracting the background intensity at 650 nm.

Figure 1 showed the visual color change of permanganate solutions at presence of different concentrations of THPS in Qurayyah AGS. Figure 2 is the permanganate absorption at 525 nm as a function of THPS concentrations. The results showed that function of THPS concentration with permanganate absorption at 525 nm is comparable in Milli-Q water and Qurayyah AGS, indicating that high salinity of Qurayyah AGS has no significant effect on the permanganate-THPS reaction. THPS can be detected as low as 2 ppmv, up to as high as 500 ppmv. Overall, the experiment concluded that the reaction of permanganate with THPS works really well in Qurayyah AGS with a dynamic detection range 2-500ppm or better.

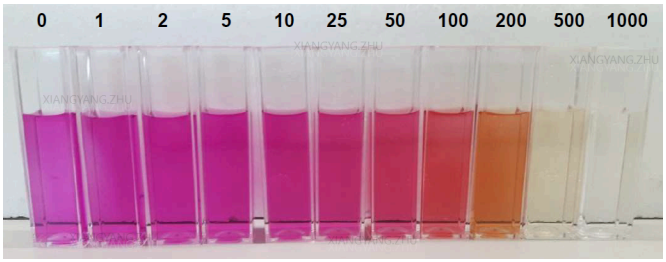

Figure 1. Color change of permanganate solution in different THPS concentrations (0 to 1000 ppmv).

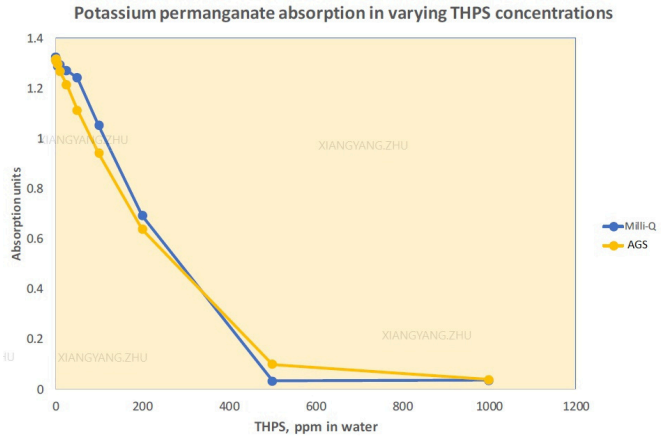

Figure 2. Permanganate absorption at 525 nm as a function of THPS concentrations in Qurayyah AGS.

Further, the permanganate-THPS reaction was validated using the commercial biocide product Duracide 9231 containing 20-40% THPS. The product was diluted in Qurayyah AGS as if it contains 30% THPS into 0, 1, 2, 5, 10, 25, 50, 100, 200, 500, 1000 ppmv of 'educated guess' THPS. Then, equal volumes of these biocide samples and 1.0 mM $KMnO_4$ in MilliQ water (pH 5.0) were mixed, and measured in the same way as described above. Figure 3 showed the normalized absorption at 525 nm as function of the 'educated' THPS concentration in Qurayyah AGS. For comparison, the series of Qurayyah AGS containing pure THPS at equal concentrations is included in Figure 3 as well.

There is small offset between pure THPS and real biocide product, both diluted in Qurayyah AGS.

The most possible explanation is that the actual THPS content of the biocide product may be higher than the 'educated' 30%, which is used in the preparation of biocide dilutions in the experiment. Nevertheless, it can be concluded that the $KMnO_4$ absorption is a very good way to measure the THPS content in seawater samples, with a linear range between 0 and 200 ppmv, a dynamic range of approximately 2-500 ppmv, and a limit of detection (LOD) of approximately 2 ppm. By increasing $KMnO_4$ concentration from 1 mM to 2 mM, the dynamic detection range of the THPS in Qurayyah AGS can be expanded to 5-1500 ppmv (data not shown).

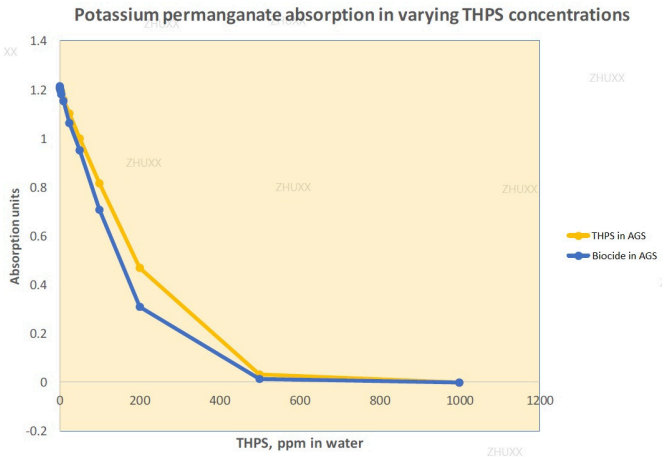

Figure 3. Permanganate absorption at 525 nm as a function of THPS concentration (pure THPS vs. real biocide product).

Validation of Detection Principle with Laboratory Mock-Up System

In order to develop the permanganate-THPS reaction into an online measurement method, a laboratory mock-up system was constructed, substituting the cuvette and lab-based absorption measurement equipment with dedicated flow-through system with reaction chamber and the absorption spectrometer.

Figure 4 is the diagram of the experimental set-up for the mock-up THPS Analyzer.

A 525 nm LED was attached to a flow cell (or Z cell) (FIAlab Instruments) for absorbance measurement with an absorption path length of 1 cm. The transmitted light is transported by a 3 mm plastic optical fiber (POF) with SMA connector to the microcontroller PCB. A Feather micro-processor platform (Arduino technology) processes the analogue signals and provides the pulses. LabView is used for the user interface and additional signal averaging. Th optical configuration for the Z-cell is illustrated in Figure 5.

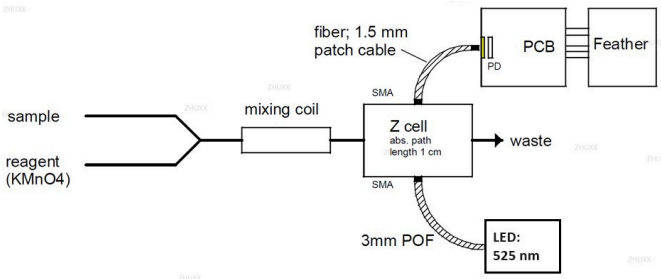

Figure 4. Experimental set-up for the mock-up THPS Analyzer. PCB - printed circuit board; POF - plastic optical fiber; SMA - SubMiniature version A.

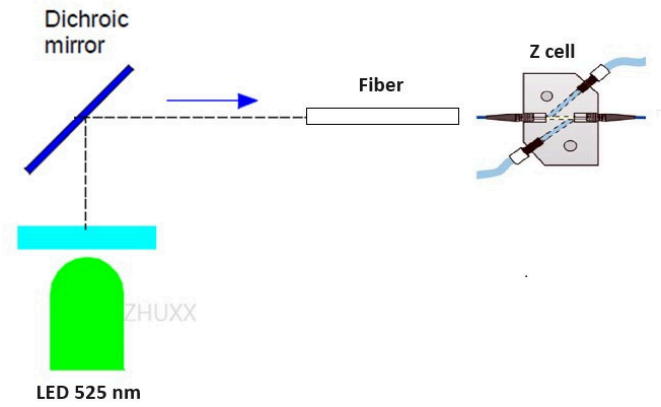

Figure 5. Optical configuration for the Z-cell.

THPS samples was prepared in Qurayyah AGS at 0, 20, 50, 100, 200, 500, and 1000 ppmv. Then, equal volumes of these THPS samples and 1.2 mM permanganate solution ($KMnO_4$·HCl, pH 5.0) were mixed in the mixing coil, and passed to the Z cell for transmission measurement at 525 nm.

Figure 6 showed the transmission measurements in the Z-flow cell of $KMnO_4$ solutions after reacting with various concentrations of THPS samples. The results showed a lower resolution at low THPS concentration range (i.e., high $KMnO_4$ concentrations) due to the lack of reference measurement at 650 nm, which was not available during the construction of the mock-up system. The resolution at low THPS concentration can be improved in a future THPS prototype analyzer by optimizing the light levels that are coupled into the flow cell and by introducing a reference channel (650 nm LED) to the current optical configuration (Figure 5). However, the results from the current setup already showed a dynamic detection range of approximately 20-1000 ppmv of THPS.

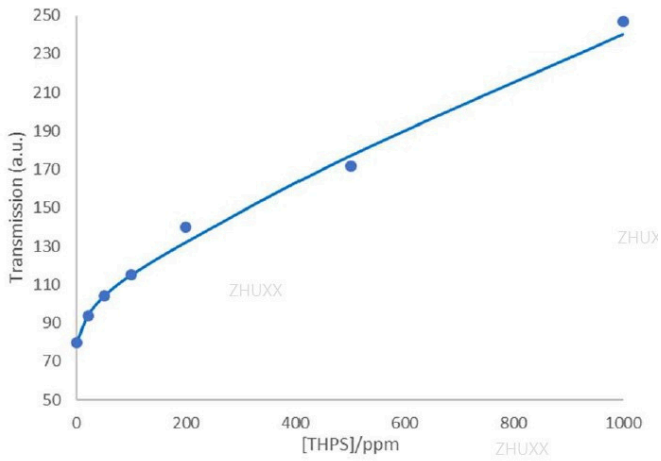

Figure 6. Measured data with the mock-up system for THPS using a single LED (525 nm).

Conclusion

This paper described and validated the permanganate-THPS reaction for the measurement of THPS and THPS-based biocide product. The method is based on absorption measurement of potassium permanganate ($KMnO_4$) solution (pH 5.0) at 525 nm for THPS detection

The method was first validated for pure THPS and THPS-containing biocide product in fresh water and Arabian Gulf Seawater (AGS) using the cuvette and lab-based absorption measurement equipment. In order to determine if permanganate-THPS reaction can be developed into an online measurement method, a laboratory mock-up system was constructed with dedicated mixing reactor, flow cell, and optical electronics. The mock-up system has showed a dynamic detection range of approximately 20-1000 ppmv of THPS prepared in Qurayyah AGS. Therefore, it is concluded that the permanganate-THPS reaction is feasible to be developed into an online analyzer for automatic detection and monitoring of THPS-based biocide with appropriate detection range. A prototype analyzer for online THPS monitoring is currently under the development using this validated permanganate-THPS reaction.

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Enhanced Hydrogen Production Using Zirconium - Based MOFs

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Abstract

The growing demand for clean and sustainable energy sources has positioned hydrogen as a promising energy carrier, particularly when produced through water electrolysis. However, the reliance on expensive noble metal catalysts like platinum hinders large-scale adoption. This study explores the use of zirconium-based metal-organic frameworks (Zr-MOFs)—specifically UiO-66, NH_2-UiO-66, and UiO-67—as cost-effective alternatives for enhancing the hydrogen evolution reaction (HER). These MOFs were synthesized via a one-pot solvothermal method and subsequently modified through cobalt electrodeposition to improve electrocatalytic performance in acidic media. Among the tested materials, UiO-67 demonstrated the highest catalytic efficiency, with further improvements observed after cobalt coating, including reduced overpotential and increased current density. Detailed electrochemical analyses revealed that cobalt integration improved electron transfer, increased active sites, and significantly enhanced HER kinetics. The findings underscore the potential of cobalt-enhanced Zr-MOFs as scalable and efficient catalysts for sustainable hydrogen production, paving the way for more affordable and environmentally friendly energy solutions.

Introduction

Recent global demand for clean and renewable energy has driven efforts to reduce dependence on fossil fuels and lower greenhouse gas emissions that contribute to climate change.[1] Among renewable energy carriers, hydrogen is a promising alternative due to its high gravimetric energy density, eco-friendliness, and recyclability. Adopting environmentally benign production techniques is essential to harnessing hydrogen as a sustainable energy carrier. Water electrolysis:

$$2H_2O \rightarrow 2H_2 + O_2$$

has emerged as a leading method for hydrogen production due to its clean and scalable nature.[2]

The electrolysis process is defined by two fundamental half-reactions (Scheme 1): the hydrogen evolution reaction (HER):

$$4H^+ + 4e^- \rightarrow 2H_2$$

occurring at the cathode and the oxygen evolution reaction (OER), occurring at the anode: $2H_2O \rightarrow O_2 + 4H^+ + 4e^-$.

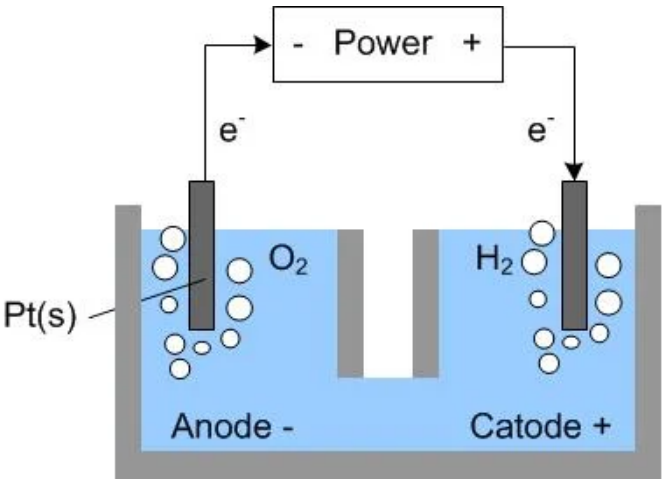

Image 1. Water electrolysis process to produce hydrogen and oxygen.

It is crucial to overcome not only the basic energy requirements of these reactions, but also additional energy barriers, which present a significant challenge in this field. [3] Currently, materials based on noble metals such as platinum,[4] iridium oxide, ruthenium oxide, [5] and gold [6] are considered among the most effective electro catalysts for hydrogen and oxygen production. However, despite their excellent performance, their high cost and limited availability hinder their widespread use. This highlights the urgent need to explore more affordable and abundant alternatives.

Metal-organic frameworks (MOFs) consist of metal nodes linked by organic linkers, offering properties such as high surface area, tunable structures, and increased porosity.[7] While these characteristics are fundamental, they are vital for boosting electrocatalytic effectiveness. Recently, the zirconium MOFs, including UiO-66 and UiO-67,[8, 9] have demonstrated their potential as effective catalysts in the water splitting process. Metal coating of these nanomaterials on the surface like Nickel (Ni) and Cobalt (Co) Copper (Cu) significantly enhances their catalytic performance.[10]

Building upon these considerations, this study focuses on the influence of cobalt electrocatalytic coatings on the hydrogen evolution performance of three zirconium-based metal-organic frameworks — UiO-66, NH_2-UiO-66, and UiO-67 — in acidic media. By enhancing catalytic efficiency and lowering overpotential, cobalt integration is expected to significantly improve HER activity, offering a promising pathway toward the development of efficient, scalable, and sustainable hydrogen production technologies.

Materials and Methods

a. Zr Based MOFs Synthesis

The UiO-66, UiO-67, and NH_2-UiO-66 MOFs were synthesized using a straightforward one-pot solvothermal method.[11] In each case, a zirconium-based precursor was combined with an organic linker (terephthalic acid or its amino-functionalized version and a biphenyl-containing acid) in a mixture of solvents (DMF and acetic acid), then heated in a sealed glass vessel at 120°C for 24 hours to promote crystallization. The mixtures were cooled, and the resulting solids were collected by filtration, thoroughly washed to remove any unreacted species or residual solvents and then dried at a moderate temperature overnight. This general process yielded crystalline MOF materials suitable for further analysis (Figure 1).

b. Electrochemical Testing

After synthesizing the materials, small amounts of each MOF were mixed with ethanol, water, and a binding agent (Nafion) to form a uniform ink. First, the different MOF mixtures were applied to carbon electrodes to test their performance (Scheme 2) . Then, the electrodes were coated with cobalt using an electrical

process to enhance their activity. All electrodes were placed in an acidic solution, and these tests helped evaluate how well each material performed in promoting the HER.

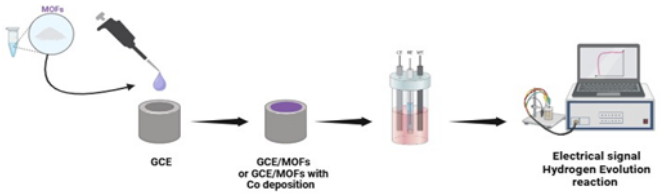

Image 2. The diagram illustrates the methodology for electrical tests of HER and OER performance

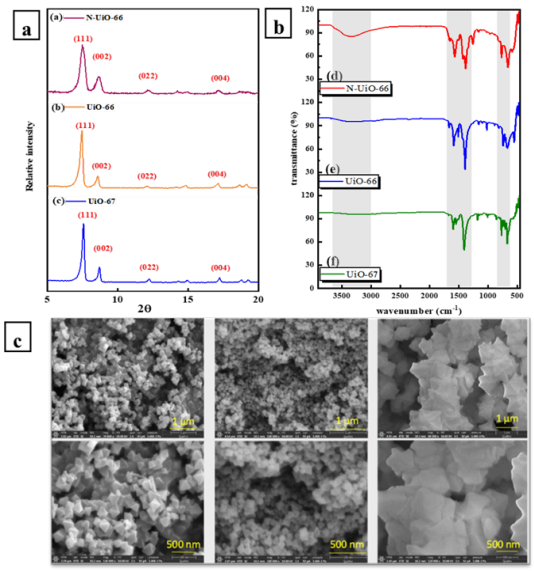

Figure 1. a. XRD Patterns, b. IR Spectra, and c. SEM Analysis of Zr Based MOFs

Results and Discussion

The electrocatalytic performance of NH_2-UiO-66, UiO-66, and UiO-67 (Figure 2 (a)) was evaluated for the hydrogen evolution reaction (HER) in 1 M H_2SO_4 using a three-electrode system. UiO-67 exhibited the lowest overpotential (-1.14 V at 10 mA cm^{-2}) and highest current density (-375 mA cm^{-2}), indicating superior HER activity due to enhanced electron transport.

Cobalt electrodeposition onto UiO-67 further improved HER performance, reducing the overpotential to -1.09 V and increasing the current density to -530 mA cm^{-2} (~43% enhancement). This improvement is attributed to increased active sites and better electron transfer.[12]

Comparative analysis of cobalt-modified MOFs (UiO-67@Co, UiO-66@Co, NH_2-UiO-66@Co) showed that UiO-67@Co had the lowest offset potential and highest current indicating the best electrochemical performance among all MOFs.

Such behavior can be attributed to the larger pore size and extended π-conjugation, which facilitates cobalt dispersion and charge mobility. The number of deposition cycles (Figure 2 (b)) also impacted HER activity. Performance improved up to 40 cycles (300 mA cm^{-2}), with no significant enhancement beyond that due to surface saturation or site blockage. Furthermore, scan rate during deposition was critical (Figure 2 (c)); a slower scan rate (0.02 V/s) yielded a more uniform cobalt layer and higher current density (~600 mA cm^{-2}), while faster rates led to less effective coatings.

Electrochemical impedance spectroscopy (EIS) confirmed a substantial decrease in charge transfer resistance after cobalt deposition of UiO-66-NH_2 reduced from ~37.5 kΩ to ~15.7 kΩ , supporting improved conductivity and HER kinetics across all MOFs (Figure 2 (d)).

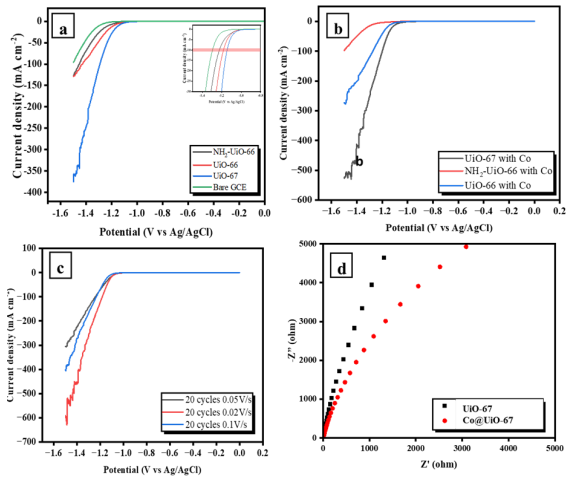

Figure 2. (a) LSV plots (b) LSV during different cycles of cobalt deposition of Zr Based MOFs, (c) LSV comparison of HER activity at different scan rates, and (d) EIS analysis before and after cobalt deposition of UiO-67.

Conclusion

This research demonstrates the effectiveness of Zr-Based MOFs in catalyzing the hydrogen evolution reaction. Among them, UiO-67 showed superior performance due to its structural advantages and electronic properties. The incorporation of cobalt via electrodeposition further enhanced the catalytic activity, particularly for UiO-67, which achieved significantly higher current density and lower overpotential. These improvements are attributed to increased electroactive sites and improved charge transfer efficiency facilitated by cobalt. Overall, the study underscores the potential of MOF-based materials, especially when combined with metal coatings, as cost-effective and efficient electro catalysts for clean hydrogen production. Future work will explore long-term stability and performance under industrial conditions to support commercialization efforts in sustainable energy technologies.

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Ydro Process®: Optimization of Sludge Production at STPs and Reduction of Associated Cost

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Abstract

The Ydro Process® offers a biotechnological advancement in wastewater treatment by harnessing naturally occurring microbial strains to significantly reduce sludge production and associated costs at sewage treatment plants (STPs). These specially selected and cultivated microorganisms exhibit resilience against common domestic inhibitors and degrade wastewater contaminants into biogas, water, and trace inorganics. When dosed daily, these facultative bacteria rapidly multiply and work synergistically with indigenous microbes, facilitating effective nutrient removal and system stabilization. The process achieves over 80% sludge reduction, complete elimination of fats, oils, and hydrogen sulfide, and substantial improvements in effluent quality (BOD, COD, TN, TP). Additionally, energy consumption, chemical use, and CO_2 emissions are considerably reduced. By maintaining oxidation-reduction potential (ORP) levels above -200 mV, the system prevents methane emissions and minimizes environmental hazards. The Ydro Process® has been validated globally for its effectiveness, offering a sustainable, scalable solution for modern wastewater management.

Introduction

The process, by which Ydro Process® microbial strains are isolated, incubated, grown and fermented, ensures that YDRO microbial products possess a natural resistance to material that would otherwise be harmful to the microbes; they are therefore able to resist inhibitors such as domestic disinfectants and cleaning solutions. The bacteria in the Ydro Process® blends produce their own enzymes in their metabolic process and treat the contaminants in the wastewater as food and convert it ultimately into biogases (e.g., CH4 and CO2), water, and a minute percentage of inorganic compounds of iron, sulfur etc., depending on the ORP conditions in the respective reaction tank.

Materials and Methods

Our bacteria are dosed to the process daily and they double every 20-30 minutes. Fresh microorganisms are added to the stream to renew the activity level of the existing. They do not replace the existing indigenous bacteria, with which they come in an operating balance. We recycle the overflow of the primary and secondary thickening to the inlet of the plant, in order select the microorganisms. (aerobic, anaerobic and facultative)

By implementing the Ydro Process® will reduce a wastewater phosphorus content from 12-15 mg/l at the plant inlet to below 1 mg/l at the plant effluent. By monitoring that concentration we can add the respective amount of iron or aluminum salts to achieve the desired level of final specification under 0.7 mg/l in the effluent. In that case an amount of sludge might have to be removed in order to keep the balance.

Furthermore, there is no methane release to the environment, since we keep the ORP level higher than -200 mV in all reactors except of course in the anaerobic digester. The phosphine concentration in the plant area is by far under the threshold concentration internationally and therefore poses no risk to humans and the environment.

We must also point out that there is a complete FOG (fat, oil, grease) removal as well as (H2S) elimination of the total system including the anaerobic digester.

Results and Discussion

By introducing naturally existing earth bio-cultures and enzymes into the sewer systems, wastewater treatment plants, or sludge storage lagoons, we achieve:
·Excess sludge reduction by more than 80% and associated costs (trucking, landfill gate fee, etc.);

- Significant improvement in plant effluent parameters (BOD, COD, SS, TN, TP, etc.)
- Significant reduction of micropollutants in plant effluent;
- Elimination of fats & oils;
- Elimination of odors/H_2S and corrosion of concrete pipelines & channels.
- Reduction in energy consumption at the aeration stage and sludge dewatering
- Gradual elimination of added chemicals.
- Carbon dioxide environmental footprint (CO_2 emissions) reduction to less than 50% of the existing.

Conclusion

The facultative bacteria survive in aerobic and anaerobic conditions, so by manipulating the ORP in the different operating tanks, we can achieve the phosphorus and nitrogen removal, which occurs with the following pathways:

The degradation process of phosphorus in the wastewater:

Organisms + Phosphate + Facultative Organisms ----→ Microbial Cells (Organophosphorus)

Microbial Cells (Organophosphorus) + Facultative Organisms----→ P2H4/PH3

The degradation process of Nitrogen in the wastewater:

1/2NH4+ (Ammonia Nitrogen) + 1/2H_2O + 1/4O_2 + Facultative Organisms ---→ 1/2NO_2 + 2e + $3H^+$

1/2NH4+ (Ammonia Nitrogen) + 1/2NO_2- + Facultative Organisms ----→ 1/2N_2 + H_2O

Author

Ahmed Zair - B.Sc. MEng - Managing Director, BIO-RAN Co. UK Based, represents the exclusive interests of the HYDROTECH ENVIROMENTAL A. Ganatsios & Co group of companies in Europe, Americas, Africa and Middle East specializing in innovative projects using advanced biotechnologies as well as biotech developments for industrial and domestic wastewater. The Ydro Process® is currently being utilized in 4 continents, more than 40 countries.

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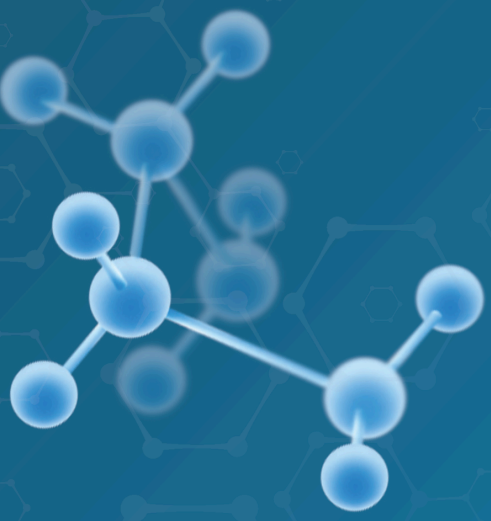

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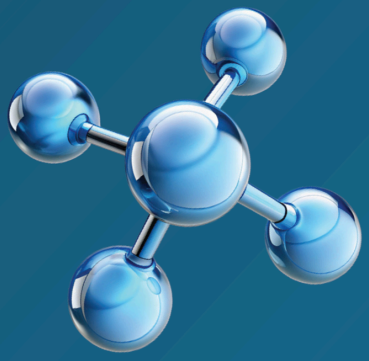

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Assessment of Quantitative Polymerase Chain Reaction Technology to Detect Sulphate-Reducing Bacteria in Crude Oil: Techniques and Findings

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Abstract

Sulfate-Reducing Bacteria (SRB) are considered a primary bacterial group responsible for Microbiologically Influenced Corrosion (MIC) in various industrial settings, including oil and gas pipelines, water treatment systems, and marine infrastructure. These bacteria play a critical role in the sulfur cycle by converting sulfate to sulfide, which contributes to the formation of corrosive hydrogen sulfide and metal sulfides. SRB also create biofilms on metal surfaces, creating a shielded anaerobic environment that promotes further growth and corrosive activities. This biofilm protects the metal surface underneath and thus hinders the effectiveness of corrosion inhibitors. Therefore, it is essential to develop effective prevention and mitigation strategies to manage MIC efficiently. The most successful approach typically involves a combination of strategies such as regulating sulfate levels, utilizing biocide treatments, restricting nutrient availability, employing corrosion inhibitors, selecting appropriate materials, performing mechanical cleaning, and implementing monitoring and early detection through laboratory assessments. The conventional NACE-TM0194 laboratory procedure; based on the most probable number theory (MPN) and serial dilution technique that requires weeks to be completed, is still followed. Thus, there is a high interest in having a rapid method with higher sensitivity and specificity to assess the presence of SRB in different environments for the control of MIC and also for taking mitigation measures. In this study, quantitative Polymerase Chain Reaction technology (qPCR); a highly reliable and sensitive molecular laboratory technique that can identify and measure the presence of bacteria in a range of industrial and environmental settings, was comprehensively evaluated to measure SRB colonies in crude oil samples. Compared to the traditional practice which can take up to 28 days to generate results, the rapid turnaround time of qPCR is a major benefit. In fact, the speed and accuracy of the evaluated qPCR-GeneCount protocol achieved through its automated and streamlined workflow, makes it possible to generate quantitative results, revealing the abundance of SRB populations in crude oil in a matter of 2-3 days maximum for a batch of 14 samples. Such rapid detection facilitates timely interventions, preventing unplanned maintenance and costly failures.

Additionally, early identification of corrosion risks supports compliance with environmental regulations, decreasing the likelihood of spills and leaks. Overall, introducing qPCR technology will significantly enhances corrosion management efficiency while promoting sustainability in crude oil production lines and associated infrastructure.

Introduction

Uncontrolled growth of microorganisms in the oil field production systems have a major negative impact on the productivity and asset integrity. Sulphate-reducing bacteria (SRB) have been found as the most troublesome group of microorganisms among all organisms involved in MIC of carbon steel and other metals used in the oil & gas industry. In addition to that, SRB contribute to hydrogen sulfide-driven reservoir souring, increased suspended solids, reservoir plugging, etc., in oil field sites (Khloud et al., 2022). To actively regulate the growth of SRB and hence prevent the impact of MIC, rapid and sensitive approaches for SRB detection are essential.

The conventional NACE-TM0194 method is still in use for detecting and estimating SRB populations. Some noteworthy limitations of this approach include its time-consuming nature, requiring lengthy incubation time for accurate enumeration. Additional challenges stem from factors such as low sensitivity, issues with media selectivity, the presence of viable but non-culturable cells, sample heterogeneity, variability, reproducibility, semi-quantification, and a lack of differentiation upon H_2S gas formation.

Sulfonation of activated carbon introduce sulfonic acid groups (RSO_2OH), enhancing the material's hydrophilicity. Additionally, this modification allows the material to hold various metals that can potentially improve the adsorption of polar organic compounds such as phenol. Metal attachment can manifest positive charges onto the surface of the material; thus, enhancing the affinity toward several organic pollutants (e.g., phenol). The primary objective of this research is to synthesize metals-modified sulfonated activated carbon, evaluate the adsorption performance for phenol removal, and compare the obtained efficiency with non-modified activated carbon. This study contributes to more effective solutions for water treatment in the oil and gas industry through understanding the underlying adsorption mechanisms.

To overcome these challenges, various protocols and techniques to quantify SRB colonies count were technically assessed. These include enzyme-linked immunosorbent assay (ELISA), Adenosine Triphosphate (ATP) bioluminescence-based testing, rapid detection pouches, and Fluorescence in Situ Hybridization (FISH). However, these methods are limited to water samples and come with drawbacks such as susceptibility to hydrogen sulfide interference, inability to differentiate SRB colonies from total bacteria, and providing only semi-quantitative results.

Consequently, we have evaluated one of the more salutary and functional molecular techniques like Real-Time Polymerase Chain Reaction, also known as Quantitative Polymerase Chain Reaction (qPCR), to be used in addition to or instead of the MPN method for SRB detection and

detection and quantification in order to get around these limitations and take into consideration a novel, practical, and effective solution.

In this work, the targeted DNA sequences of SRB species were isolated and measured using the qPCR-GeneCount protocol in crude oil samples. By amplifying and detecting the distinct genetic signatures; particular to the targeted SRB populations, this protocol and which makes use of the quantitative PCR principle has been fully assessed.

Materials and Methods

Following the manufactures' manual and guidelines, the qPCR-GeneCount assay kit was used to specifically identify and quantify SRB species in crude oil samples. The present study adheres to the worldwide reference standard (AMPP TM21465-2024 Molecular Microbiological Methods-Sample Handling and Laboratory Processing) in both protocol and applied process.

Sterile glass containers to collect one-liter volume of crude and water samples were used for this microbiological analysis. To maintain the integrity of the collected samples and to avoid any population shifts due to microbial growth during the transfer from sampling points to the laboratory, samples were maintained at low temperatures (4 °C or below).

Major steps of the analysis workflow include the sample pre-treatment; to extract the water phase from the crude oil sample, DNA preservation, DNA purification and extraction, SRB assay tubes preparation; which contains the primer complementary to the targeted SRB sequence beside the dNTPs and a fluorescent intercalating dye, qPCR reaction setup, qPCR quantification, data analysis and data interpretation. It bears mentioning that a batch of 14 samples can be loaded at a time in the thermocycler device to perform the qPCR experiment: thus, obtaining results in 2-3 days maximum, with some pre-analytical and post-analytical steps involved.

In the thermocycler device, SRB genetic sequences will be exponentially amplified from a small initial amount of the targeted DNA through a programmed thermal cycling profile (41 cycle). A sequence of temperature-cycling phases is used during the qPCR reaction to assist with the various stages of DNA copies amplification. Theses stages include thermal denaturation, primer annealing, and DNA extension, which are considered three major steps that make up one complete cycle of the qPCR thermal profile. After the last cycle, a final extension step ensures complete synthesis of all DNA strands.

To track and monitor the amplification process, a fluorescence channel is employed in tandem with DNA amplification. The fluorescent dye employed for SRB detection is called EvaGreen. It attaches itself to the minor groove of double-stranded DNA and, when stimulated by a certain light wavelength, releases green fluorescent light. The examiner can then measure the amount of SRB present in the unidentified samples by measuring the fluorescence intensity, which rises as the number of DNA copies increases during the amplification process.

The generated qPCR data is computed by comparing the fluorescence levels after each cycle to a pre-loaded standard curve and the initial DNA amount. The provided quantitative results and which is directly related to SRB abundance in samples is then reported as cells/mL.

Each analysis conducted to measure the unknown targeted samples shall be in conjunction with running a known concentration of positive control (10^6 copies/RXN) and a no template negative control (NTNC). By using these reference controls, it can be confirmed that the qPCR-GeneCount assay is operating as expected and is producing accurate findings.

Results and Discussion

More than 50 samples of water extracted from crude oil were tested to verify the accuracy and reliability of the abovementioned evaluated methodology. The following table (Table-1) shows the acquired data from analyzing several crude oil samples. All tested samples were below instrument detection limit (10^2 cells/mL).

From one point of view, examining and referring to the historical trends of SRB growth in crude oil was one of the approaches used to cross-check and validate the gathered data. In fact, all the presented data in table-1 which indicate below detection limit readings (BDL) were compared to previous results reported from an identified and relevant geographical region, where similarly, only null results were reported by another technical entity for the same tested sampling points of crude oil.

Table 1. Results of SRB in Crude Oil by qPCR-GeneCount; *BDL: Below Detection Limit

GOSP	Line Tag	Analysis Date	SRB Reading (Cells/mL)
A	Positive Control	5/5/2024	PASS
	TR-1		*BDL
	TR-2		BDL
	TR-3		BDL
	TR-4		BDL
	TR-5		BDL
	TR-6		BDL
B	Tr-1		BDL
	Tr-2		BDL
	Tr-3		BDL
C	Positive Control	5/21/2024	PASS
	TR-1		BDL
	TR-2		BDL
	TR-3		BDL
	TR-4		BDL
	TR-5		BDL
	TR-6		BDL
D	TR-1	5/29/2024	BDL
	TR-2		BDL
	TR-3		BDL
	TR-4		BDL
	TR-5		BDL
	TR-6		BDL
	TR-7		BDL
	Positive Control		PASS

Moreover, samples from one of the sea water injection wells were tested for enumerating SRB colonies before the biocide injection (table-2), and show a growth of SRB that is commonly reported at similar levels, before the treatment and inhibition of bacterial replication.

Table 2. Detection of SRB in sea water samples by qPCR-GeneCount

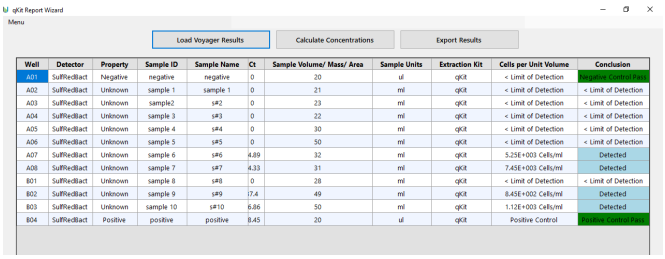
PLANT	Sampling Point	Analysis Date	SRB Reading (cells/mL)
H-GOSP	A	5/28/2024	1740
	B		1510

In addition, number of crude oil samples were spiked and measured under a variety of conditions and concentration levels of SRB cells that extracted from positive culturing media vials and Rapid Detection Pouch techniques. qPCR-GeneCount assay has detected the elevated, moderate,

and low concentrations in consistency with the approximate growth scale as shown in the below table.

Table 3. Spiked samples consistency with an expected range (order of magnitude of 10)

Expected concentration for the extracted SRB (cells/mL)	Readings obtained by qPCR-GeneCount SRB Assay (cells/mL)
> 1,000,000	1,610,000
100,000 - 1,000,000	662,000
10,000 - 100,000	16,500 / 16,600 / 17,200
100 - 10,000	845 / 1,120/ 1,400/ 4,800 / 5,250 / 7,450



Well	Detector	Property	Sample ID	Sample Name	Ct	Sample Volume/ Mass/ Area	Sample Units	Extraction Kit	Cells per Unit Volume	Conclusion
A01	SulfRedBact	Negative	negative	negative	0	20	ul	qKit	< Limit of Detection	Negative Control Pass
A02	SulfRedBact	Unknown	sample 1	sample 1	0	21	ml	qKit	< Limit of Detection	< Limit of Detection
A03	SulfRedBact	Unknown	sample2	s#2	0	23	ml	qKit	< Limit of Detection	< Limit of Detection
A04	SulfRedBact	Unknown	sample 3	s#3	0	22	ml	qKit	< Limit of Detection	< Limit of Detection
A05	SulfRedBact	Unknown	sample 4	s#4	0	30	ml	qKit	< Limit of Detection	< Limit of Detection
A06	SulfRedBact	Unknown	sample 5	s#5	0	50	ml	qKit	< Limit of Detection	< Limit of Detection
A07	SulfRedBact	Unknown	sample 6	s#6	8.89	32	ml	qKit	5.25E+003 Cells/ml	Detected
A08	SulfRedBact	Unknown	sample 7	s#7	8.33	31	ml	qKit	7.45E+003 Cells/ml	Detected
B01	SulfRedBact	Unknown	sample 8	s#8	0	28	ml	qKit	< Limit of Detection	< Limit of Detection
B02	SulfRedBact	Unknown	sample 9	s#9	7.4	49	ml	qKit	8.45E+002 Cells/ml	Detected
B03	SulfRedBact	Unknown	sample 10	s#10	6.86	50	ml	qKit	1.12E+003 Cells/ml	Detected
B04	SulfRedBact	Positive	positive	positive	8.45	20	ul	qKit	Positive Control	Positive Control Pass

Image 1. A displaying model for one batch of spiked and positively detected samples results reported as Cells/mL

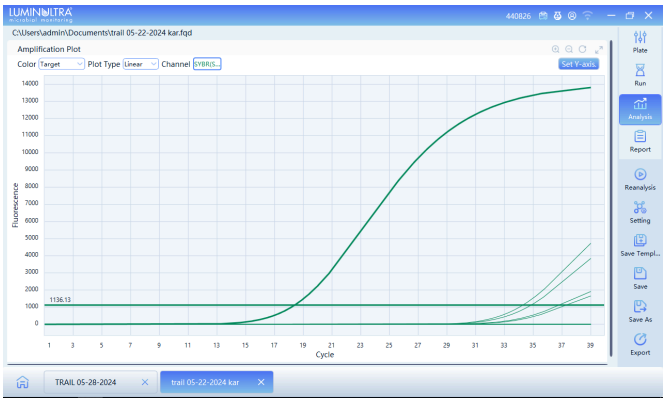


Image 2. A displaying model for one batch of spiked and positively detected sample's amplification plot to illustrate the increase in fluorescence intensity

Conclusion

The suitability and effectiveness of qPCR-GeneCount technology for the identification and enumeration of SRB species in crude oil have been thoroughly investigated and successfully validated. The presented new strategy demonstrates several advantages over traditional methods, which often involve lengthy time frames.

Key benefits include the high specificity, pre-designed and validated assay, automation, pre-formulated reagents, quantitative measurement capabilities, H2S interference mitigation, and rapid identification. Such features position qPCR-GeneCount technology at the front of other solutions in the market. Moreover, the input of the applied technology will help corrosion engineers in assessing the potential risk of SRB-related issues and forecast MIC trends over time.

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Green Catalytic Oxidation of Benzyl Alcohol to Benzaldehyde Using Ginger and Pomegranate Peel Extracts: A Selective and Sustainable Alternative to Traditional Metal Catalysts

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Abstract

This study explores the green catalytic potential of Zingiber officinale (ginger) and Punica granatum (pomegranate peel) extracts in the selective oxidation of benzyl alcohol to benzaldehyde—a reaction of industrial importance in the fragrance and pharmaceutical sectors. The catalytic activity of the plant extracts was evaluated under mild conditions (60°C, 30 minutes) in the presence of hydrogen peroxide as an oxidant. Gas chromatography–mass spectrometry (GC-MS) confirmed the formation of benzaldehyde with a conversion rate of 71.4% ± 2.3% for ginger extract, outperforming pomegranate extract (59.8% ± 2.7%) and approaching the efficiency of manganese dioxide catalysts (75.6% ± 1.9%). Kinetic analysis revealed a lower activation energy for plant-based systems compared to conventional metal catalysts. FTIR and UV-Vis spectroscopy supported the mechanistic role of phenolic and flavonoid components in catalysis. Thermal stability and reusability were assessed via TGA and multi-cycle tests, demonstrating the environmental and practical viability of these green alternatives. This study affirms the potential of plant-derived materials as eco-friendly catalysts for selective oxidation reactions.

Introduction

Benzaldehyde is a key intermediate in the production of fragrances, pharmaceuticals, and fine chemicals. Traditional oxidation of benzyl alcohol to benzaldehyde often relies on metal-based catalysts, which pose environmental and health hazards due to toxicity and limited reusability. The increasing demand for sustainable chemical processes has encouraged the development of eco-friendly catalytic systems. Plant-based catalysts, particularly those rich in polyphenols and flavonoids, have shown promise in mediating oxidation reactions via hydrogen peroxide activation. This study investigates the green catalytic performance of ginger and pomegranate peel extracts in oxidizing benzyl alcohol under mild conditions. These extracts are selected for their availability, antioxidant properties, and known catalytic potential in redox reactions. By comparing their efficiency with that of manganese dioxide, this research aims to highlight a sustainable alternative that aligns with green chemistry principles.

Materials and Methods

Materials

Benzyl alcohol (99.5%), hydrogen peroxide (30% w/w), ethanol (absolute), and manganese dioxide (analytical grade) were obtained from Sigma-Aldrich. Fresh ginger (Zingiber officinale) roots and pomegranate (Punica granatum) peels were collected from local organic suppliers, thoroughly washed, and used for extract preparation.

Preparation of Plant Extracts

Both ginger and pomegranate peels were sliced, oven-dried at 50°C for 48 hours, and ground into a fine powder. Each powder (20 g) was extracted in ethanol (200 mL) under magnetic stirring at 300 rpm for 24 hours at ambient temperature. The mixtures were filtered through Whatman No. 1 filter paper, and the filtrates were concentrated using a rotary evaporator at 40°C. Extracts were stored in amber bottles at 4°C.

Oxidation Procedure

The oxidation of benzyl alcohol was conducted in 50 mL round-bottom flasks. A typical reaction mixture consisted of 5 mmol benzyl alcohol, 10 mL distilled water, and 2 mL of either plant extract. Hydrogen peroxide (3 mmol) was added dropwise, and the mixture was stirred magnetically at 60°C for 30 minutes. A comparative reaction using manganese dioxide (0.05 g) as a traditional catalyst was also conducted under the same conditions.

Analytical Methods

The reaction products were analyzed using Gas Chromatography–Mass Spectrometry (GC-MS, Agilent 7890B GC with 5977A MSD, HP-5MS column), with a 1 µL injection volume under splitless mode. FTIR spectra were recorded using a Shimadzu IRTracer-100 spectrophotometer in ATR mode over the 4000–500 cm^{-1} range. UV-Vis spectra were recorded with a Jasco V-730 spectrophotometer from 200–400 nm to monitor product formation.

Thermal Analysis and Reusability

Thermal stability of the catalysts was assessed by Thermogravimetric Analysis (TGA) using a PerkinElmer TGA 8000 from 25°C to 600°C at a heating rate of 10°C/min under nitrogen. Reusability tests were performed over three catalytic cycles, with recovery via centrifugation and ethanol washing after each use.

Kinetic Studies

The reaction was repeated at three different temperatures (40°C, 50°C, 60°C) to determine rate constants. The Arrhenius equation was used to calculate activation energy (Ea) from plots of ln(k) versus 1/T (K).

Results and Discussion

Catalytic Performance

The catalytic efficiency of the plant-based extracts was assessed through the oxidation of benzyl alcohol to benzaldehyde. Ginger extract achieved a conversion rate of 71.4% ± 2.3%, while pomegranate peel extract yielded 59.8% ± 2.7%, both under mild conditions (60°C, 30 min). Manganese dioxide, used as a benchmark catalyst, resulted in 75.6% ± 1.9% conversion. The enhanced activity of ginger extract is attributed to its higher content of phenolic and flavonoid compounds, which are known to activate hydrogen peroxide and facilitate electron transfer reactions.

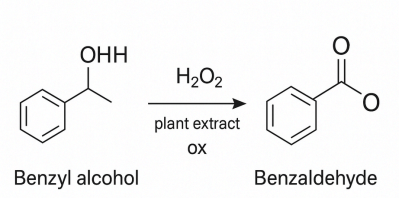

Figure 1. Illustrates the catalytic oxidation of benzyl alcohol using the plant-based extracts, emphasizing the simplicity and eco-friendliness of the system.

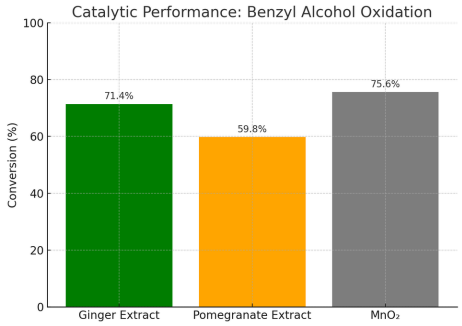

Figure 2. Demonstrates the comparative performance of all tested catalysts, highlighting ginger's close performance to the conventional MnO_2.

Spectroscopic Confirmation

The product formation was confirmed using FTIR and UV–Vis spectroscopy. FTIR analysis revealed a sharp C=O stretching band at ~1720 cm^{-1}, indicative of aldehyde formation, alongside the attenuation of the O–H stretching band near ~3300 cm^{-1}. UV–Vis spectra showed increased absorbance at ~252 nm, corresponding to $\pi \rightarrow \pi^*$ transitions in aromatic aldehydes. In the ginger system, absorbance rose from 0.35 to 0.61, confirming product formation. These findings are consistent with prior studies on plant-based oxidation systems (Singh et al., 2022, ACS Sustainable Chem. Eng., 10(3), 1456–1464).

Mechanistic Insight

The reaction mechanism likely involves the activation of hydrogen peroxide by phenolic hydroxyl groups in the extracts, leading to the formation of reactive peroxy species that oxidize benzyl alcohol.

This pathway aligns with mechanisms observed in other flavonoid-rich catalytic systems. Figure 3 presents a mechanistic depiction of the proposed steps.

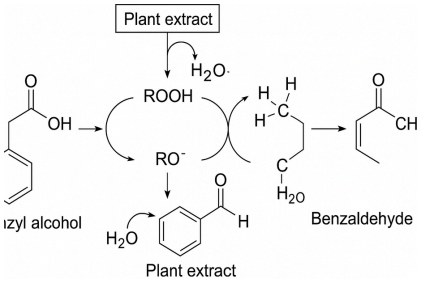

Figure 3. Proposed mechanistic pathway showing the role of phenolic compounds in activating hydrogen peroxide and facilitating alcohol oxidation.

Kinetic Analysis

Experiments conducted at 40°C, 50°C, and 60°C allowed the construction of Arrhenius plots and the estimation of activation energies. The derived Ea values were:

- Ginger extract: 42.8 kJ/mol
- Pomegranate extract: 48.5 kJ/mol
- MnO_2: 38.1 kJ/mol

These results indicate that the plant-based systems offer competitive thermodynamic profiles. Figure 4 provides a comprehensive summary of catalytic performance, activation energy, thermal stability, and reusability for the tested systems.

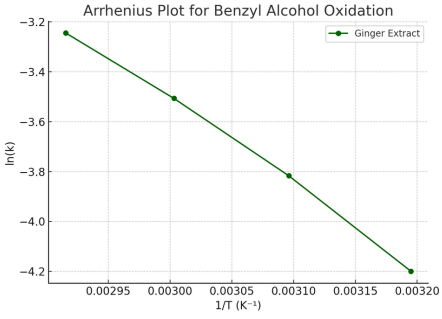

Figure 3. Summary table of catalytic systems: conversion efficiency, activation energy, thermal stability, and reusability.

Thermal Stability and Reusability

Thermal gravimetric analysis (TGA) confirmed that both ginger and pomegranate extracts remain stable up to ~180°C, well above the operating temperature. In reusability trials, the ginger extract retained over 85% of its catalytic activity after three cycles, reinforcing its suitability for repeated use in green oxidation processes.

Catalyst Comparison and Summary

Figure 5 summarizes key parameters of the catalytic systems, including conversion efficiency, activation energy, thermal stability, and reusability. Ginger extract demonstrated the most favorable balance of performance and sustainability.

Catalyst	Conversion (%)	Activation Energy (kJ/mol)	Thermal Stability	Reusability (After 3 Cycles)
Ginger Extract	71.4 ± 2.3	42.8	High	~90%
Pomegranate Peel Extract	59.8 ± 2.7	48.5	Moderate	~83%
MnO_2	75.6 ± 1.9	38.1	High	Not applicable

Figure 5. Comparative infographic summarizing key performance indicators and sustainability metrics for all catalysts.

Economic Assessment

A rapid economic comparison revealed that plant-derived catalysts cost only $0.15–0.25 per gram, compared to $1.20/g for manganese dioxide (analytical grade). Additionally, the green catalysts generate no metal waste, reducing environmental burdens and eliminating hazardous disposal requirements.

Industrial Applicability

The simplicity, cost-effectiveness, and high selectivity of these systems make them ideal candidates for use in small-to-medium-scale production of benzaldehyde derivatives, including flavoring agents, fragrance intermediates, and pharmaceutical precursors.

These systems also have potential for adaptation in continuous flow reactors, making them scalable and industrially viable.

Conclusion

This study demonstrates the viability of ginger and pomegranate peel extracts as green, metal-free catalysts for the selective oxidation of benzyl alcohol to benzaldehyde under mild conditions. The ginger extract, in particular, exhibited a high conversion efficiency (71.4%) that closely rivals conventional MnO_2 catalysts, while offering significant environmental and economic advantages. Spectroscopic analyses confirmed the selective formation of benzaldehyde, and kinetic evaluations revealed favorable activation energies for both plant systems. The extracts also showed excellent thermal stability and reusability, affirming their potential for repeated applications. The simplified reaction conditions, low cost, and sustainability of the plant-derived catalysts position them as attractive alternatives for industrial applications in fine chemical synthesis. Future work may explore their integration into continuous-flow processes or their functionalization for broader substrate scopes.

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Incorporating Biomolecules into MgCaFe-LDH for Enhanced Phosphate Adsorption

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Abstract

Phosphate pollution poses a serious threat to aquatic ecosystems due to its role in triggering harmful algal blooms and disrupting biodiversity. In this study, novel biomolecule-incorporated MgCaFe-Layered Double Hydroxide (LDH) composites were synthesized using a co-precipitation method to enhance phosphate adsorption from wastewater. Vitamin D and aspartic acid were selected as representative biomolecules based on their functional roles in phosphate regulation in biological systems. The LDH composites were synthesized with the incorporation of each biomolecule and tested for phosphate removal efficiency using batch adsorption experiments under controlled conditions. Results showed that biomolecule modification significantly enhanced the adsorption performance of LDH materials, with the aspartic acid modified composite achieving a removal efficiency of 91.4% after 24 hours. Vitamin D modified LDH also demonstrated substantial improvement, indicating effective interaction between the biomolecule's functional groups and phosphate ions. This approach introduces a sustainable and eco-friendly method for reusing expired biomolecules while improving water treatment efficiency. The findings support the potential of biomolecule-functionalized LDH materials as high-performance adsorbents for phosphate remediation in line with environmental sustainability goals.

Introduction

Water pollution remains a persistent global challenge, with phosphate contamination being among the most critical threats to aquatic ecosystems. Excessive phosphate levels primarily originate from industrial discharges, agricultural runoff, and domestic wastewater. When released into water bodies, phosphate serves as a nutrient that accelerates the growth of algae, often resulting in harmful algal blooms (HABs). These blooms not only deplete dissolved oxygen but also block sunlight, disrupt aquatic biodiversity, and render water unsafe for both human use and marine life. The urgency to remove phosphate from wastewater is therefore both an environmental and public health imperative. Among the wide array of treatment methods available, adsorption has proven to be one of the most effective, economically viable, and environmentally friendly techniques. The search for high-performance adsorbents has thus become a

central focus in environmental chemistry and water treatment research [1].

Layered Double Hydroxides (LDHs), particularly those containing Mg, Ca, and Fe, have attracted considerable attention due to their unique structural features and ion-exchange capabilities. These materials consist of positively charged layers of metal hydroxides, which are balanced by interlayer anions such as NO_3^- or CO_3^{2-}. This structure allows for high surface area, controllable composition, and remarkable anion exchange properties. More recently, attention has shifted to combining LDHs with organic or biological molecules to enhance their performance in selective adsorption. Among such biomolecules, Vitamin D (cholecalciferol) stands out due to its pivotal role in phosphate metabolism in the human body. This biological insight has inspired a novel approach: integrating Vitamin D into LDH composites to potentially enhance phosphate adsorption from water by mimicking biochemical phosphate regulation mechanisms [2].

The present study explores the synthesis of various biomolecule-modified MgCaFe-LDHs using a co-precipitation method, with a focus on Vitamin D and aspartic acid as representative biomolecules. The aim is to investigate how these molecules, known to interact with hydroxyl groups and regulate ionic behavior in biological systems, may influence the structure, surface chemistry, and overall adsorption efficiency of LDHs. The idea stems from the dual need to enhance water purification technologies and to repurpose expired pharmaceutical and biochemical products such as Vitamin D supplements. The hypothesis is that incorporating biomolecules into LDH structures will enhance their performance in removing phosphate from water through improved binding interactions, increased functional groups, and structural modulation of the LDH matrix [3].

Materials and Methods

The synthesis of MgCaFe-LDH composites was achieved through a well-established co-precipitation method. Three metal salts calcium nitrate, magnesium nitrate, and iron nitrate were dissolved in deionized water. The pH of the mixture was adjusted to 9.5 ± 0.5 using 2 M NaOH while stirring continuously. In separate trials, the same procedure was followed with the incorporation of different biomolecules: Vitamin D, aspartic acid, and sodium nitrate, each introduced individually to form distinct LDH composites. The biomolecules were expected to interact with the hydroxyl groups of the LDH structure during the co-precipitation process, either through hydrogen bonding or via direct complexation with the metal ions.

The resulting solutions were stirred at 60°C for approximately 20 hours, allowing sufficient time for the crystalline LDH phases to form and stabilize with the integrated biomolecules. After the reaction period, the precipitates were filtered, washed, and dried. The dried powders were then crushed and stored for adsorption testing. For phosphate removal experiments, 30 mg of each synthesized composite was added to 40 mL of phosphate solution with an initial concentration of 25 mg/L. The mixtures were shaken at 240 rpm at 30°C for time intervals of 24 and 48 hours. After contact, the solutions were filtered and analyzed for phosphate concentration using standard

test kits, following protocols that ensure accuracy and repeatability.

This experimental setup was designed to evaluate not only the removal efficiency of phosphate ions by different composites but also to assess the influence of contact time and biomolecular type on adsorption performance. The data obtained from these experiments were compiled into tabular and graphical formats to compare the removal efficiency across different conditions and biomolecular modifications. Special attention was given to composites with Vitamin D due to its potential functional role in enhancing adsorption via interaction with phosphate through similar pathways observed in biological systems.

Results and Discussion

The successful synthesis of MgCaFe-LDH and its biomolecule-modified variants was confirmed visually by the formation of consistent and homogenous powder materials. Upon testing, the parent LDH (without any biomolecule) showed moderate phosphate removal efficiency after 24 hours, which improved slightly after 48 hours. However, the addition of biomolecules significantly enhanced the performance. Among the modified composites, the LDH incorporating aspartic acid demonstrated the highest phosphate removal rate, achieving a remarkable 91.40% after just 24 hours as shown in Table 1. This outperformed both the parent LDH and other biomolecule-LDH composites under the same conditions.

Table 1. The effect of contact time using a parent MgCaFe-LDH and different biomolecules-LDH composites on the phosphate removal at 30° C for 24 and 48 hours (the initial concentration is 25mg/L).

LDH Composites	24 hours	48 hours
MgCaFe-LDH-NO_3^-	3.67 mg/L	1.77 mg/L
MgCaFe-LDH-vit.D	4.54 mg/L	2.28 mg/L
MgCaFe-LDH-ASP	2.15 mg/L	<1.60 mg/L

Vitamin D modified LDH composites also exhibited substantial improvements in phosphate adsorption efficiency. The improved performance can be attributed to the presence of functional groups in Vitamin D, such as hydroxyl groups, which may interact with phosphate anions through hydrogen bonding or nucleophilic substitution. These interactions may facilitate stronger bindings of phosphate ions on the LDH surface, enhancing the composite's adsorption capacity. Additionally, Vitamin D's polycyclic structure might have contributed to better dispersion or stabilization of the active sites within the LDH matrix. While not surpassing the performance of aspartic acid composites, the Vitamin D-modified LDH still demonstrated considerable potential and introduces a new perspective in using expired biomolecules for environmental remediation.

Data analysis revealed a general trend of increased phosphate removal with longer contact time, although in some composites the majority of adsorption occurred within the first 24 hours, indicating rapid uptake. The phosphate removal mechanism is likely a combination of surface adsorption, anion

exchange within the interlayer spaces of LDH, and specific binding interactions introduced by the incorporated biomolecules. The presence of these biomolecules likely increased the affinity of the composite surface for phosphate ions by introducing additional binding sites or altering the charge density across the LDH layers [4]. The variation in removal efficiency between the different biomolecules also highlights the role of molecular structure and functional group chemistry in determining adsorption performance.

The practical implications of these findings are significant. The use of biomolecule-modified LDH composites for phosphate removal not only enhances performance but also provides a sustainable solution for reusing pharmaceutical waste. Expired Vitamin D supplements, which would otherwise contribute to pharmaceutical pollution, can be repurposed as functional additives in water treatment technologies. Moreover, the environmental friendliness of the LDH-biomolecule system makes it suitable for a range of applications, including fish tanks, aquariums, aquaculture facilities, and environmental cleanup projects [5]. This fits well with sustainability goals such as Saudi Arabia's Vision 2030, which emphasizes environmental protection and waste minimization.

Conclusion

In conclusion, this study demonstrates that incorporating biomolecules such as Vitamin D and aspartic acid into MgCaFe-LDH composites significantly enhances their phosphate adsorption capacity. Through a straightforward and cost-effective co-precipitation synthesis, various LDH-based composites were successfully prepared and tested. The incorporation of biomolecules was found to play a pivotal role in modifying the surface chemistry and structural properties of LDH, ultimately influencing its performance in removing phosphate ions from aqueous media.

Among the composites tested, aspartic acid modified LDH achieved the highest phosphate removal efficiency, while Vitamin D modified LDH showed promising results that warrant further investigation. The findings not only confirm the potential of biomolecule-LDH composites in water treatment but also introduce an innovative way to recycle expired pharmaceutical compounds for environmental benefit. This dual advantage enhancing water purification and reducing pharmaceutical waste offers a unique and sustainable solution for tackling phosphate pollution in freshwater and marine systems.

Future work will focus on optimizing synthesis parameters such as biomolecule loading, LDH composition, and pH conditions. Additionally, long-term stability and reusability studies will be essential to determine the practical applicability of these composites in large-scale treatment facilities. Further exploration of other biomolecules and their synergistic effects when combined with LDH structures may lead to even more efficient and environmentally friendly adsorbents. Ultimately, the approach presented in this work has the potential to contribute meaningfully to the development of advanced materials for sustainable water purification.

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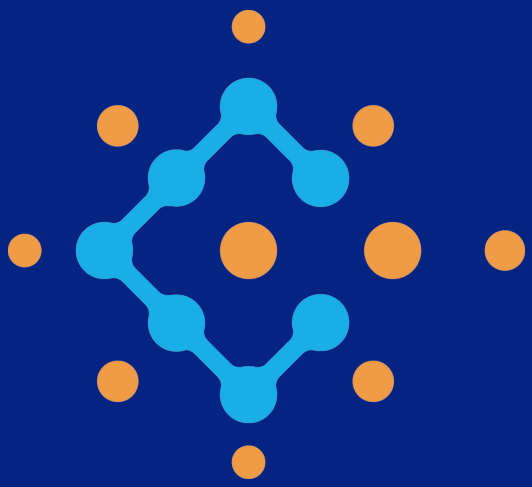

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