POTENTIAL INTEGRATIONS OF MICROWAVE TECHNOLOGY IN PROMOTING THE SUSTAINABLE PRODUCTION OF POLYHYDROXYALKANOATES: A MINI REVIEW

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Abstract: Microwave (MW) irradiation offers rapid, selective heating at the molecular scale and underpins two advanced methods—microwave-induced oxidation (MIO) and microwave-assisted extraction (MAE). MIO is already deployed industrially, especially in wastewater treatment, where it achieves high removal efficiency without generating secondary pollutants. This review summarizes recent MIO advances in wastewater remediation and highlights its emerging role in sludge pre-treatment that enhances the anaerobic digestion and boosts volatile fatty-acid (VFA) yields. VFAs can in turn serve as cost-effective substrates for microorganisms that accumulate polyhydroxyalkanoates (PHAs). We also detail MAE as a green, efficient alternative to conventional PHA recovery. Taken together, current evidence reveals multiple integration points at which MW-based techniques can lower cost and improve the sustainability of the entire PHA value chain.

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Introduction

In an attempt to reduce the dependence on petroleum-based plastics and mitigate issues related to plastic pollution, the rapid development of bio-based and biodegradable polymers has emerged as one of the most potential solutions. In this context, polyhydroxyalkanoates (PHAs) have particularly received attention the most thanks to the promising mechanical and thermal properties and being exceptional in biodegradability compared to traditional plastics.[1] By adjusting the constituent monomers, PHAs, moreover, can be modified into different co-polymers, providing diverse properties that can meet technical requirements in both engineering and household applications. Therefore, PHAs are expected to thrive, being a potential alternative to minimize the overconsumption of conventional plastics.

In general, PHAs are intracellular polyesters accumulated by various bacteria under imbalance nutrient conditions.[2] Accordingly, PHAs can be produced in a sustainable manner by bacterial fermentation. The commercialization of PHAs, nevertheless, is hindered by its high production costs, which can be attributed to both upstream and downstream processes. Precisely, the pricey upstream stage is mainly ascribed to the use of expensive conventional substrate, including refined sugar- and food-grade feedstocks, which can constitute up to 30 – 50% of the total production cost.[3] On the other hand, the steps of recovery and purification in PHA extraction, which consumes a considerable amount of chemicals, are responsible for the costly downstream operation.[4]

In this regard, several studies have been done thoroughly, in which advanced technology of microwave (MW) irradiation could be a useful means to solve the aforementioned problems. In fact, MW technology has

been well-known for the use of electromagnetic waves to transmit stable and integrity analog signals in telecommunications and wireless networking. Electromagnetic waves, moreover, can penetrate dielectric material, causing internal oscillations and friction between polar molecules, which eventually convert the absorbed microwave into heat.[5] This heating mechanism is determined to be superior in terms of heating rates, energy efficiency, being selective and excellent in controllability, which not only turns MW technology into a compelling alternative for conventional heating methods but also a transformative tool of oxidation in numerous fields, including chemical synthesis,[6] chemical extraction,[7] food processing,[8] environmental remediation[9] and waste/wastewater treatment.[10]

Accordingly, the main goal of this mini review is to present the potential integration of MW technology in PHA production. This information is provided by a discussion on applying strategies of microwave-induced oxidation and microwave-assisted extraction to facilitate the PHA production from waste materials together with the more ecofriendly methods of PHA extraction. Lastly, challenges and future research avenues are envisaged, approaching closer to the more sustainable and economically feasible production of PHAs.

The basis of Microwave (MW) technology and its unique mechanism of heating

Standing between radio waves and infrared waves, microwaves are a part of electromagnetic spectrum with corresponding wavelengths of 1 mm to 1 m and frequencies of 0.3 to 300 GHz, respectively.[11] These band features have provided MW with a great information-carrying capacity, ensuring the preservation of data during transmission. Consequently, MW

technology has been playing an important role in modern wireless communication technologies.

Moreover, MW irradiation provides rapid, selective heating that has broadened its industrial applications. As an electromagnetic radiation, MW has the characteristic of wave-particle duality that allows it to interact with substances in diferent ways, concurrently inducing heat generation by two principle mechanisms of dipolar polarization and conduction.[12] In fact, the heating mechanism of MW technology is highly attributed to the effect of electromagnetic field on charged particles of exposed substances, especially lossy dielectric materials. Under electromagnetic field of MW, polar molecules of lossy materials are originally rearranged into the field direction and subsequently resonate with the continuously alternating frequencies. The dipoles, however, cannot cope with the rapid change in the electric field direction. Nevertheless, the vigorous rotation of dipole molecules conversely creates intense frictions and collisions inside the molecular structures which eventually ends up to internal heat generation.[13]

In this aspect, the heating rate of lossy materials greatly depends on its dissipation factor, which is calculated by the following equation:

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'}$$

where ε'' is the relative loss factor that characterizes the ability to dissipate electric energy and ε' is the dielectric constant of the material, presenting the capacity of electric energy storage. Accordingly, lossy materials with high ε'' are more readily heated by MW irradiation, posing another tempting feature of selective heating.[12]

On the other hand, electrical conductors such as metals or graphite are a typical reflector of MW. However, under the impact of MW, free charge carriers of conductor materials are polarized, inducing electron flows

and simultaneously heating the object surfaces by conduction mechanism of electrical resistance. Lastly, good insulators (e.g., quartz, porcelain, ceramics, etc.) are more "transparent" due to the absence of charge carriers, permitting MW energy to penetrate without any losses or dissipations for heat production.[13] Insulator materials, nevertheless, can also be heated by the support of facilitator (for instance, magnetite or silicon carbide), which is initially heated by the MW and then transfer to the insulator.

Microwave-Assisted Strategies for Sustainable PHA Production

The unique heating mechanism of MW irradiation underlies two advanced techniques – microwave - induced oxidation (MIO) and microwave-assisted extraction (MAE) – that accelerate reactions, enhance selectivity and save energy.[5] The advantages of MW-related technologies have highly promoted the treatment of low value organic waste streams and the establishment of less-solvent extraction methods, increasing thus the efficiency of resource use. Accordingly, this section will provide information about MIO and MAE techniques, with the emphasis on improving the sustainbability and cost-feasibility in PHA production.

Microwave-induced oxidation: principles and development

The development of microwave-induced oxidation (MIO) is conducted based on the molecular-level heating of microwave irradiation. After absorbing MW energy, the heat generation from molecule oscillations and collisions reaches an intense level, which is able to oxidize the components direcly or indirectly. Depending on the sample size, depth penetration and relative loss factor, the thermal process could be homogenous or heterogeneous and selective, creating localized "hot spots"

that accelerate the oxidation process.[7] Therefore, MIO is an useful tool applied widely in many fields with environmental section is currently one of the sectors that benefited the most (Figure 1).
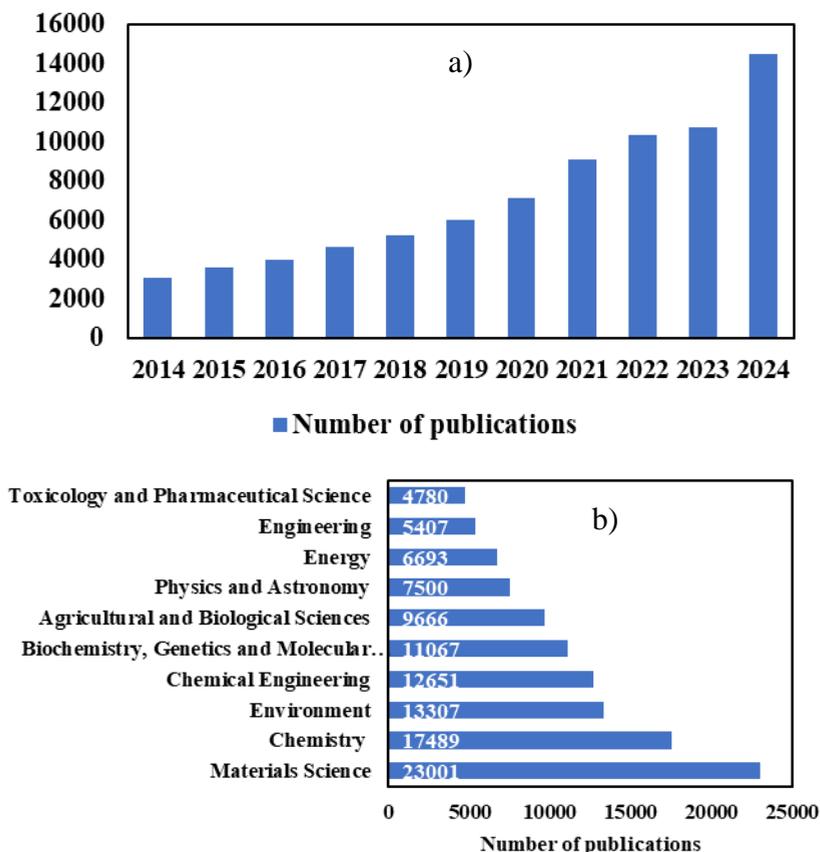

Figure 1. Scientific articles related to microwave-induced oxidation (MIO) from 2014 to 2024 a) and Number of MIO publications classified in fields b).

Current environmental applications of microwave-induced oxidation

In general, many volatile and semi-volatile compounds (e.g., pyridine, nitroglycerin, and ammonia-derived species) are highly reactive and may form more toxic derivatives through secondary reactions in the environment. Therefore, MIO has been applied as an effective technology to degrade these compounds and mitigate such risks. By using MIO

technology, the removal rate of volatile compounds reached 93% - 100% in lab-scale experiments, using MW power of 225 – 750W, in 5 – 10 seconds.[14,15] In the case of total ammonia, the application of MIO was successfully scaled up to pilot scale, achieving an elimination efficiency of 85% at initial concentrations up to 11,000 mg/L.[16]

On the other hand, MIO technology can combine with oxidant and catalyst materials to promote the degradation of complex pollutants. The addition of MW-induced oxidation materials increases both the absorption of MW energy and the formation of "hot spots", which subsequently enhance the generation of active substances as hydroxyl radicals ($\bullet OH$) and superoxide radicals ($\bullet O_2^-$) in aqueous solution.[5] Free radicals, afterwards, induce further reactions with recalcitrant pollutants to form mineralized products. Aromatic compounds, for example, are the typical complex contaminants that commonly exist in wastewaters. In this regard, dyeing effluent is one of the richest sources of aromatic pollutants, posing numerous difficulties for conventional treatment methods of absorption, chemical oxidation, electrolysis, etc. Dye substances, however, were found to be effectively degraded by MIO technology with the support of MW-induced catalyst. In a study of Zhang, et al.,[17] 50 mg titanium dioxide nanoparticles supported activated carbon (TiO_2/AC), was used to assist the treatment of methyl orange (50 mg/L) under the MW irradiation at 750W, rendering the removal efficiency of 95%. Similar outcomes were obtained in studies by Riaz and Ashraf [18] and Wang, et al.,[19] showing a removal rate of 90 % and 99 % in the degradation of orange G and methyl blue, respectively. Interestingly, at a high concentration of Rhodamine 6G dye of up to 5000 mg/L, the performance of catalyst-supported MIO technology was still efficient.[20]

Aquaculture, chemical-derived, pharmaceutical wastewaters with concentrated antibiotic and phenolic compounds are other challenging effluents that now can be productively handled by catalyst-combined MIO technology. The mechanism of MW-induced catalytic degradation was strengthened in a study of Liu, et al.,[21] depicting the important role of active substances in the decomposition of antibiotic of tetracycline (TC). High-performance catalyst of manganese oxide on carbon nanotubes (MnO/CNTs) revealed a removal rate of TC of 185.7 mg/g (TC/catalyst) in 10 min. Other TC derivatives of TC hydrochloride and chlortetracycline were efficiently decomposed by MW irradiation combined catalyst of $ZnFe_2O_4$ and nano-Fe_3O_4 on carbon nanotubes (nFe_3O_4/CTs), showing a degradation efficiency of 86% and 90%, respectively.[22,23] Similarly, phenolic compounds can be well-disintegrated under catalyst-assisted MW irradiation. p-Nitrophenol, for instance, was decomposed at a level of 94.70% by MW-assisted $NiCo_2O_4$-$Bi_2O_2CO_3$ within 1 min.[24] In a study of Qiu, et al.,[25] pristine bismuth oxide (Bi_2O_3) was excited by MW energy to form electron-hole pairs on the surface, where water molecules were converted into hydroxyl radicals to oxidize p-nitrophenol, providing a removal efficiency of up to 99.74%. The similar pattern of degradation was also found in the study of Yin, et al,[26] which decomposed 99.6% of p-nitrophenol by using Mn_2O_3/AC supported 400W MW energy.

Besides, MIO technology also showed promising results in treating other wastewaters, including coke-containing wastewater,[27] landfill leachate,[28] pesticide wastewater[29] and refinery wastewater,[30] etc. Compared to conventional methods which are costly, land consuming, advanced operating conditions, slow and low removal rate, MIO technology offers many advantages, making it advisable for industrial-scale development.

Microwave-induced oxidation in waste valorization and the

potential integration in polyhydroxyalkanoate production

In addition to being an effective method for wastewater treatment, MIO has also emerged as a promising approach for waste valorization, with wastewater sludge (WWS) as a representative example. WWS is generally known as a semi-solid by-product generated mainly during the primary and secondary stages of wastewater treatment plants. Untreated sludge contains high moisture (90–98%), elevated biochemical oxygen demand (BOD) and pathogenic microorganisms.[31] The high BOD is primarily attributed to organic carbon and essential nutrients such as nitrogen and phosphorus, which can be further converted into biogas, energy and fertilizers through anaerobic digestion (AD), incineration or composting, respectively.[32]

Among these options, AD is considered the most mature and cost-effective technology for WWS stabilization. However, complex organic macromolecules and recalcitrant bacterial cell walls significantly limit the hydrolysis step, thereby restricting overall digestion efficiency.[13] In this context, MIO represents a compelling pretreatment technology capable of enhancing AD performance. Through superheating and the generation of reactive radicals at localized "hot spots", microwave irradiation disrupts sludge floc structure, solubilizes organic matter and stabilizes complex substances, which subsequently improves biodegradability.[33] Eskicioglu, et al.[33] reported increased soluble proteins and enhanced biodegradability following MW pretreatment, resulting in approximately 20% higher biogas production. Compared with conventional heating, MW-acclimated inoculum yielded 16% higher biogas production.[34] Moreover, the effectiveness of MIO was maintained at high sludge concentrations up to 5.4% TS (w/w), and was in some cases superior to treatment at lower sludge contents.[35]

When combined with thermophilic AD, MW pretreatment even achieved biogas yields up to 106% at reduced sludge retention times.[42] Additional studies consistently confirm these benefits, highlighting MIO as an efficient pretreatment to improve AD performance.[36-40]

Despite these clear improvements, the economic feasibility of biogas remains constrained because profit margins for biomethane are relatively low compared to the required capital and operational expenses. Consequently, interest has shifted from methane recovery toward the production of volatile fatty acids (VFAs), which offer higher economic value as platform chemicals.[41] VFAs are key intermediates produced during the acidogenic phase of AD, which exhibits higher conversion rates, process flexibility and more stable yields than the methanogenic stage.[42] To maximize VFA production, methanogenesis is intentionally suppressed, typically through heat shock or chemical inhibitors.[43] Chemical inhibition provides advantages such as straightforward implementation, precise control and scalability, yet raises concerns regarding cost, environmental toxicity and residual accumulation. Heat shock is generally more selective and environmentally benign, although it still requires substantial energy input.

Owing to its rapid and selective heating capability, MIO presents a viable alternative for suppressing methanogenesis while simultaneously enhancing sludge solubilization. Mehdizadeh et al.[37] showed that treating waste activated sludge above 80 °C effectively prevented biogas production. Complete suppression of methanogenesis was also observed by Özön and Erdinçler[43] using persulfate-assisted MW irradiation. In another study, Liao et al.[44] combined MW with hydrogen peroxide and sulfuric acid, achieving up to 96 % solubilization of tCOD and recovering 25 % of sCOD as acetic acid. Beyond chemical oxidants, MIO can be integrated with ultrasonic

irradiation and other advanced oxidation processes to further increase sludge bioavailability.[45,46] Collectively, MIO and its derivatives function as powerful WWS pretreatments that enhance solubilization, improve biodegradability and suppress methanogenesis, thereby steering AD toward higher VFA yields.

VFAs, whether as individual compounds or mixtures, serve as versatile chemical building blocks for applications in biomaterials, cosmetics, fine chemicals, food, petrochemicals and pharmaceuticals.[47] Among these applications, polyhydroxyalkanoate (PHA) synthesis is particularly attractive. VFAs act as key precursors in microbial PHA biosynthesis, enabling potential yields of up to 78%. Considering the widespread deployment of anaerobic digesters worldwide and the demonstrated ability of MIO pretreatment to improve VFA production, VFAs generated from MIO-assisted AD offer a cost-effective and sustainable substrate supply, thereby enhancing both the economic viability and environmental sustainability of PHA production.

Microwave-assisted extraction in facilitating the recovery of polyhydroxyalkanoates

Besides the possibility of supporting upstream process, MW technology can also play another important role in downstream process of PHA recovery. As an intracellular product, the extraction of PHAs is quite challenging, requiring to disrupt and/or lyse the whole bacterial cell wall, followed by purification steps to obtain the final products with high degree of purity.[48] This can be conducted in different ways, including physical, chemical and biological methods with various outcomes. Among them, the chemical extraction using solvents is the most effective one, providing the

highest yield and purity. Solvent extractions of PHAs, however, commonly consume huge amounts of chemicals with chlorinated- and organic compounds, for example, chlorofom, sodium hypochlorite, methanol, ethanol and acetone, etc. are the popular ones.[51] The conventional recovery of PHAs, therefore, has been also known as a costly and less environmentally friendly process, partly contributing to the high-cost production of PHAs.

In this respect, the support of MW energy with extraordinary heating characteristics can alleviate the drawback of solvent extraction. In fact, the combination of MW technology and solvent extraction has been known as microwave-assisted extraction (MAE) which has been found to surpass the conventional methods in terms of processing times, solvent usage reduction and extraction yield.[49] With high-moisture content of up to 70%, the water inside bacterial cells can be rapidly evaporated under MW irradiation, inducing high-internal pressure that results in cell rupture. This, in turn, facilitates the access of chemical solvents to the microbial cells by the larger contact area between the solid and liquid phases, leading to the exceeding release of desired compounds.[50] This mechanism has been widely applied in the extraction of antioxidants and lipids from plants and microalgae, respectively, which has been described thoroughly in numerous theoretical and experimental studies.[51-54] The theory of cell rupture, moreover, was strengthened in a study of Qi, et al.[55] and Badwaik, et al.,[56] which observed a massive change and destruction in the morphology of leave samples under scanning electron and light microscope after using MW exposure-supported solvent extraction.

Regardless the huge potential, the application of MAE in PHA extraction is relatively scarce. It was initially used only in the last step of

drying to achieve the desirable moisture content of 0.5% in PHA granules for adequate post-processing.[57] Considering the efficiency of MW-assisted drying without decreasing PHA yield, Akdoğan and Çelik[58] utilized the MW technology in dehydration stage, prior to solvent extraction. In this study, the use of MW-assisted dehydration not only increased the overall volumetric productivity by 1.5 times, but also significantly reduced the energy consumption (0.09 MJ compared to 44.1 MJ of lyophilization process). In another study of Balakrishna Pillai, et al.,[59] a chloroform-free extraction method was conducted by using EDTA-microwave assisted cell lysis. This combination completely disrupted the *E. coli* cell wall, providing comparable results to that of sodium hypochlorite treatment without damaging the molecular weight of extracted PHAs. Accordingly, Bocaz-Beltrán, et al.[60] upgraded the use of MAE in PHA recovery from mixed microbial cultures. A duplicated 2-level factorial model was designed to evaluate the effects of processing factors including time, solvent ratio (chloroform/methanol), temperature and solvent/biomass ratio on extraction efficiency. Compared to conventional heating extraction, MAE demonstrated itself as a time-saving method by a higher extraction rate while the yield and efficiency were ensured. In this study, the use of MAE was done directly on wet microbial biomass which can potentially skip the lyophilization step and subsequently reduces the recovery cost. MAE, moreover, was coupled with ultrasonic waves to create intense turbulence in a fluid which improves the process of mass transfer.[61] With a stronger energy, this rapid protocol accelerated the digestion process using sodium hypochlorite, increasing the yield and quality of extracted PHAs in terms of thermal resistance and crystalline properties. According to the promising results obtained, MAE is deemed to be an extraction method of choice in

recovering PHAs that highly fulfills criteria of technical aspect, economic feasibility and environmentally friendly. A schematic overview highlighting the role of MAE within the PHA recovery pathway, together with its interaction with upstream biomass processing, is presented in Figure 2.
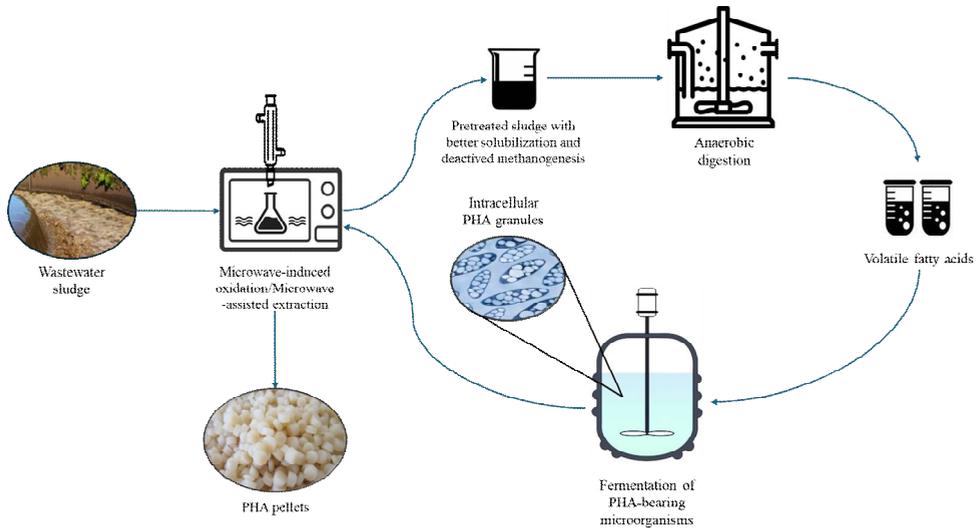

Figure 2. Conceptual integration of microwave-assisted extraction (MAE) within the downstream recovery of polyhydroxyalkanoates (PHAs).

Future prospects

The future prospects of microwave-induced oxidation and microwave-assisted extraction are greatly promising which highly lies in the ability to increase the overall efficiency, reduce energy consumption, minimize solvent use and shorten extraction time compared to conventional methods. As a result, these technologies are posed to be an innovative approach to address the environmental and industrial challenges, particularly in waste valorization to harmonize with the current concept of circular economy.

Nevertheless, in order to fully exploit the benefits of MW technology, some of following factors need to be thoroughly considered.

Firstly, it should be noticed that the effectiveness of MW system tends to increase at high energy input and long exposure time, due to the amplification in contaminant decompositions. The condition of which, however, possibly leads to the release of extra heat and higher viscosity from excess evaporation of water.[62] Therefore, the future development of both MIO and MAE should focus on the optimization of the operatory conditions, including power, frequency and exposure time, to maximize the processes of oxidation and extraction, at the minimum energy consumption and minimum byproduct formation. Heat recovery, furthermore, is another essential aspect that should be taken into consideration to benefit cost reduction and energy savings. This can be done by integrating the system of heat exchangers and/or thermal storage unit to leverage the excess heat for further ancillary purposes of preheating, drying or combined heat and power systems. Research into hybrid systems combining MIO and MAE with other advanced oxidation processes could create synergistic effects, expanding its versatility to more complex materials. Additionally, innovations in reactor designs with the main direction on improving heat distribution, material compatibility, real-time monitor, ease of operation and solvent recovery should be considered to enhance the consistent and reproducible outcomes. Lastly, life cycle assessments and cost-analysis studies are essential to ensure sustainability and economic feasibility, thereby enabling the scalable deployment of MW technologies in accordance with industrial standards.

Conclusions

The research on MW technology has recently obtained practical achievements from which benefit numerous industrial sectors. The application of MW-based techniques, therefore, is forecasted to be kept

widely used and developed, increasing research interest and tremendous potential in new study direction. In this review, MW technology was introduced to support the production of PHAs in both upstream and downstream processes. By coupling MIO and AD, wastewater sludge can be effectively valorized into economical substrates for the fermentation of PHA-bearing microorganisms. While MIO can improve the recovery of PHAs with a great edge in efficiency and environmentally benign over conventional methods. This concept is believed to have a great impact on the commercialization of PHAs, which yet requires further analysis on optimization, system integration, cost-effectiveness and life cycle assessment in the future.

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