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Analysis Of Recent Research In Solketal Synthesis Toward Circular Economy : A Review

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ABSTRACT. Indonesia's growing biodiesel production has caused a large surplus of crude glycerol, which becomes both an economic and environmental problem. The study present a systematic chemical engineering perspective on how glycerol can be transformed into solketal, a compound that can act as a biofuel additive and green solvent. The discussion is based on a systematic reading method of twelve research papers published between 2018 and 2024, specifically discuss the development of catalysts related to this process. The contrasting performance between catalyst efficiency and its industrial implementation become a concern. It shows Zeolites and MOF catalyst exceed other types of catalysts by using process intensification in laboratory. But in contrast, their stability lower when associated with impurities found in feedstock, followed by heat-related problems. On the other side, simple and cheaper catalysts such as acid-treated clays still show steady activity even in less clean feedstocks, making them more realistic for larger scale operation. The review also points out that improving catalyst lifetime and using new process approaches like microwave heating are important to make the process more economical. Overall, this study suggests that creating catalysts which can resist impurities together with better process design is necessary to make glycerol utilisation more sustainable and in line with Indonesia's circular economy plan.

1. INTRODUCTION

Known as one of the largest palm oil-producing countries, Indonesia has managed to contribute large-scale biodiesel to the industry, albeit with a global imperative towards renewable energy sources[1]. Biodiesel, mainly produced from process called transesterification, a reaction involving vegetable oils or animal fats, accounts for the delivery of its byproduct, glycerol, to be widely abundant.

For years, glycerol production contribute about 10% from biodisel production[2]. This explain both a challenge and an opportunity due to depressing market prices. Also, it pose environmental impact if not managed .

According to their structure, glycerol functions as a C_3 platform chemical ($C_3H_8O_3$) that can be converted into valuable products using various mechanism. For example, uplifting its original purpose, not only as abundant as it is, but it also can be reacted with acetone to result high value solketal. Thus, the environmental necessity to manage waste and supporting circular resource utilisation[3]. Aside from that, crude glycerol is great concern due to its small demand for their market.

The conversion into solketal is a very critical component of developing a sustainable and economically viable biorefinery sector. Thus, utilising locally available glycerol aligns with Indonesia's circular economy goals-initiated by the Indonesian Ministry of Industry[4]-by converting waste into a valuable chemical, yet it also reduces reliance on petrochemical feedstock.

The advances of solketal production is noticeably becoming the interest of many researchers across the globe, according to recent study[5], solketal can be derived from various processes. That being said, every method of solketal synthesis is dependent on locally available raw material from country to country. Making it adaptable and forming many different structural mechanisms in each area. This context-specific adaptability highlights the need for more essential systematic literature review into regionally optimised solketal production pathways.

2. MATERIALS AND METHODS

The This study employs a Systematic Literature Review(SLR) in order to identify and synthesize relevant scientific literature, mainly focusing on Solketal synthesis. As previously studies[6,7] have suggested, the review should be filtered into several steps:

2.1 Literature Search and Identification

The search was performed using *Publish or Perish(PoP)* software to index publications from three major academic databases, namely, Crossref, Semantic Scholar, and Google Scholar. The search was confined to several research papers, articles, and proceedings published between 2018 to 2024. The following keywords were used for the query: *"(solketal OR "fuel additive"), (glycerol OR glycerin), (acetalisation OR ketalisation), catalyst".*

The initial queries resulted in a total of 454 potential literatures, with 186 papers identified from Crossref, 68 papers from Semantic Scholar, and 200 papers from Google Scholar.

2.2 Study Selection Process

The indexed articles were then subjected to a filtering/screening process based on predefined exclusion and inclusion criteria. This process includes the screening of titles and abstracts.

The exclusion criteria will likely exclude papers that (a) only mention solketal as a chemical without discussing its synthesis, (b) focus only on the application of solketal, or (c) review articles that did not present new experimental data.

In contrast, the inclusion criteria will likely include every study focused on the direct synthesis of solketal, particularly from glycerol, and investigated reaction parameters and catalysis. After screening, the literatures from Crossref shrinks into smaller number of 11 papers (N1), Semantic Scholar with 6 papers (N2), and Google Scholar with 10 papers (N3).

2.3 Eligibility and Justification for Sample Size

The Following the initial screening process, the 27 articles were advanced to the full text review and confirmed to be 13 studies that met the suitable inclusion criteria. Thus, 13 final papers will be analyzed for further discussion on the advancement of solketal synthesis through the years between 2018 to 2024. Figure 1 shows the selection and methodology process of this systematic literature review.

The relatively final sample size (N=13) reflects the specialised and nature of solketal synthesis research. Mosf of excluded papers either (a) lacked sufficient experimental detail to enable comparative analysis, (b) focused on distinct conversion pathways (i.e., glycerol to acrolein, glycerol to glycerol carbonate) that fell outside the scope of specific solketal synthesis). Furthermore, the inclusion criteria requiring explicit catalyst characterisation data and quantitative performance metrics narrowed the potential inclusion lists.

Despite the limitation, the total size of selection (N=13) provides sufficient studies for comparative analysis and identification of technological trends, even though the absolute number may limit stastitical power of certain quantitative comparisons.

3. RESULTS AND DISCUSSION

Thirteen significant journals and papers were discovered as a consequence of the SLR method's literature search. The following research findings, shown in Tables 1 and 2, were used as references in this study.

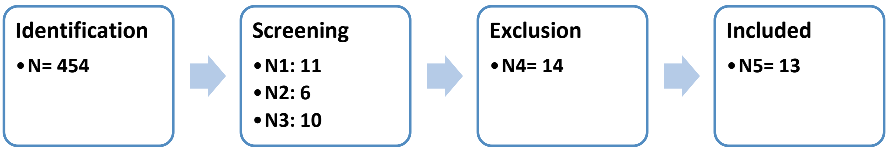

Figure 1. Selection and Article Screening

Table 1. Selected Result Articles

No.	Authors	Summary
1.	[8]	Using Basolite F300 (MOF) was able to convert 84.33% pure glycerol at 50°C
2.	[9]	Using $[HMIm]_3[PW_{12}O_{40}]$@MOF-Fe at 30°C within 10 min able to convert 99% glycerol
3.	[10]	Using Amberlite IR 120 Na gave conversion only to 38.94% at 55°C along with addition step.
4.	[11]	Using Amberlyst-36 able to result in 86.61% Conversion at 50°C
5.	[12]	Using Amberlyst-36 exceeds even higher conversion of 88.19% at 50°C with some slight modification
6.	[13]	Using Indion 225 Na able to covert 28.50% glycerol at 55°C`
7.	[14]	Using ionic liquid catalyst able to yield reactant uo to 86% at 30°C within 30 min of reaction.
8.	[15]	Using CO_2 as switchable catalyst able to reach 61% conversion at 118°C
9.	[16]	Using modified clay to convert glycerol up to 73% conversion at 50°C
10.	[17]	Using WO_3/SAPO-34 Zeolite reach up to 96.70% yield at 60°C.
11.	[18]	Using Sulfated Metal Catalyst able to reach 99.30% glycerol of conversion at 70°C
12.	[19]	Using carbon catalyst from waste tire able to reach 90% at 40°C
13	[20]	Using microwave-assisted method with SAFACAM carbon catalyst able to reach 98.10% conversion of glycerol at 70°C within 10 minutes

3.1 Catalytic Landscape for Solketal Synthesis

The current literature shows a clear performance in catalytic system, in which advanced, self-prepared catalyst often perform better compared to comercially available catalysts. This is related with the ability to design the physicochemical properties, such as controlled porosity, adjusted acid dencity, and also better stability under reaction conditions. On the other hand, the commercial catalyst sometimes give a different performance caused by their material limitations rather than the actual ceiling of the glycerol acetalisation process

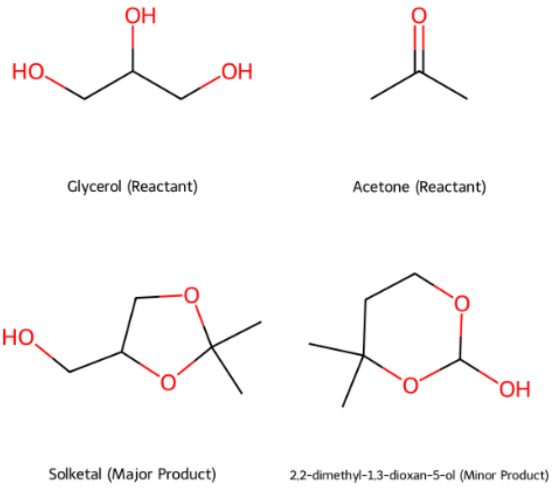

Figure 2. Reactant and Products of Glycerol Acetalisation

3.1.1. *Commercial Ion-Exchange Resins as Benchmark Catalyst.*

Sulfonic acid ion-exchange resins are usually applied as reference to provide a baseline for evaluation novel system in glycerol acetalyzation. Among them, Amberlyst-36 catalyst as described in [11,12] has shown good catalytic activity, achieving glycerol conversion above 85% under optimised reaction conditions. Because of that, it often used as a representative benchmark in many studies. In continuous-flow systems, it can also reach a conversion rate between 85% and 97% with pure wet glycol, maintaining high selectivity and stability[21,22]

Nevertheless, the performance of other catalyst, Amberlite IR 120 Na gave a final acetal product with only 38,9% purity, whilst proven to be followed by a distillation process afterward [10]. The distillation should improve separation efficiency. Likewise, [13] stated that Indion 225 Na can reach a maximum glycerol conversion of only 28.5%, which is far lower than the industrial standard. From this comparison in table 2, the conversion performance of commercial resin catalyst show dependancy on their textural and structural feture. This may because of their porosity, crosslinking density, acid site affinity and sulfonation's degree.

Table 2. Comparison of Resin Catalysts

Catalyst	Temp	Mol Ratio	Time (min)	Conversion (%)
Amberlyst-36[11]	50	1:6	120	86.61
Amberlyst-36[12]	50	1:4	180	88.19
Indion 225 Na[13]	55	1:5	180	28.50
Amberlite IR 120 Na[10]	50	-	90	38.94

To conclude, this analysis underline commercial sulfonic acid resins are valuable as basis that they don't represent the highest catalyst performance. Second, the large variation in results canon the necessity of catalyst design notably for their morphology, acidity, and the balancing of hydrophobic-hydrophilic to achieve better reactivity and selectivity.

3.1.2. Performances of Advances Heterogeneous Catalyst

The development of laboratory solid acids produced an advancement of heterogeneous catalyst that pivot on reactivity and selectivity. These materials can be designed to control of their surface acidity, porosity, and their thermal stability. This led to favorable environments for ketalisation can be achieved. Modifying catalyst properties will open the pathway to comprehend structure-activity relation in terms of governing catalytic performance.

Metal-Organic Frameworks (MOFs)

MOFs are a class of crystalline materials notable for their high surface area. Basolite F300 is an example that demonstrate significant catalytic activity. Previous research has reported that it reach about 83.3% glycerol conversion even when only 1 wt% catalyst was added[8]. The kinetic analysis of their study revealed an activaton energy around 15.7 kJ/mol. This suggests that acetalisation route is driven more by thermodynamic favorability than by higher kinetic barrier.

In reviewing the literature, Authors found that MOFs still have major issue for their stability under some operational conditions. Their structure based on previous studies[8], [9] can be compromised in hydrotermal environment if the reaction were conducted in a prolonged time. It may be the disorder of crystalline structure led to the collapse under such such condition.

Modified Clays and Zeolites

It is noteworthy to mention that some natural element can be utilised as promising catalyst. Namely, clay if chemically modified can give around 73% glycerol conversion even when crude, unpurified glycerol was used[16]. Based on the study, the compromise of this activated clay concept has tolerance for impurities like water, methanol, and salts. In contrast, other catalysts likely deactivate more sensitive than MOFs.

Also, advanced zeolite-based frameworks are capable of achieving much higher efficiency. i.e. the WO_3/SAPO-34 system has been reported to deliver solketal yields up to 96.70%[17]. This significance phenomenon of combination of weak, medium, and strong acid sites led to the synergistic effect for stability and reactivity. These results highlights how careful structural engineering on zeolitic frameworks can push catalytic performance very close with the theoretical maximum.

From the material metallurgy perspective, clays and zeolites reflects how the framework is created and stabilised. Zeolite synthesis often needs calcination, insertion of heteroatoms and very controlled crystallisation in order to make the required pore size and acidity. This require significant energy and costs, and even when scaled-up. In reverse, clay are abundant aluminosilicates and only require simple acid activation. It is such a benefit due to there is no need for heavy metallurgical process. In that sense, the catalyst preparation become a crusial factor so that zeolites can deliver more yield. In other hand, clays shows how minimal treatment can deliver more suitable option for industrial application.

Sulfated Mixed Metal Oxides

Within the class of solid acids, sulfated mixed oxides have emerged as some of the most promising catalysts. This is because they have exceptional lift such as better surface acidity, tunable properties, and high catalytic performance in various reactions, including solketal acetalisation. They are considered effective due to their link to density and strength of surface acid sites, which are enhanced by sulfation and the synergistic effects of combining multiple metal oxides.

Zinc-aluminium-zirconium oxide (SO42-/ZnAl2O4-ZrO2 or SZZ) reported by [18] had achieved near-quantitive conversion up to 99,3% along with 98% solketal yield. This is due to the morphological structure and cristalinity, the higher and more area will surpass the catalytic performance compared to analogues with less optimal structure or lower acidity [23–25].

Table 3. Comparison of Sulfated Metal Catalysts

Catalyst System	Key application	Performance Highlights	Source
$So_4^{2-}/CeO_2\text{-}SiO_2$	NH3-SCR (Nox reduction)	High SO2 resistance, strong acidity	[23]
$So_4^{2-}/SnO_2\text{-}ZrO_2$	Alkylation, oxidation	High conversion, improved stability	[24]
$So_4^{2-}/FeO_3\text{-}SiO_2$	Organic synthesis	Enhanced Brønsted/Lewis acidity, reusable	[25]

Despite the higher activity, complex synthesis procedure and the need for precise control over composition and structure can hinder the scalability of sulfated mixed oxides for industrial deployment. Thus, advances in preparation methods will likely hold the key to unlock the full potential of these catalysts.

Carbon-Based Catalysts from Sustainable Sources

The conversion of waste into catalyst is in line with the circular economy principle. Within 30 minutes of reaction, a pyrolytic carbon black from end of tires (rCB)[19] has shown remarkable initial activity that leads to almost 90% conversion of glycerol. Another material from renewable feedstock also gain 98,1% efficiency of converting glucose into sulfonic acid reported in the other work[20]. This alternatives established less conversion time and advances in reaction technology. However, the long term stability of these catalyst need to be tuned properly. By recycling, it can lead to accelerated catalytic decline due to the presence of heavy oligomeric byproducts. Moreover, active group leaching often results in blockage during multiple runs of the process. It is very concerning to implement engineering strategies to optimise their durability to make carbon-based catalyst more practical than before.

3.1.3. Novel Systems for Homogeneous Catalysis

In recent years, heterogenous catalysts dominate glycerol acetalisation research due to their reusability and ease of strength. For example, heteropolyacids immobilised on silica or zeolites can reach up to 99% conversion and 97% selectivity at room temperature, analogous to sulfonated graphene and carbon-based catalysts that deliver rapid yet high-yield reactions with good reusability[3,26,27]. Homogeneous catalysts eliminate internal diffusion limitations by operating it in the same phase as reactants. It also lessens mass transfer resistance that is often problematic in porous solids. This enables highly efficient contact between reactant and a catalytic sites, potentially improved rate of the reaction under mild conditions.

Ionic Liquids(ILs) type of catalyst is considered alternative bacause of their tunable phycochemical properties. It also have minimal vapor pressure effect. For example, TPPT has remarkable catalytic activity. It was reported on the study[14] showing the experimental can deliver 86% solketal yield at 30°C in only 30 minutes. This high activity followed by the excellent operational stability, maintaining performaces in catalytic reaction over ten consecutive cycle wihtout efficiency loss. This marks notable breakthrough that is rarely observed in homogenous systems for the level of reusability. In basic priciple of separation, the product was achieved to be drawn through liquid-liquid extraction. TTPT has higlighted how careful molecular design of ILs can provide both high efficiency and operational practices.

Switchable catalyst, another proposed analogues for solketal synthesis[15], involves the use of those catalyst that can be easily deactivated or removed on demand. This concept utilise the use of pressurised CO_2 as green witchable catalyst. Unden high pressure condition, CO_2 will reacts with water in the mixture from carbonic acid in situ, then followed by catalysation of glycerol in acetalisation process. Once the reaction is complete, a depressurisation mechanism release the CO_2, removing the catalytic species without leaving residue in the process .

This phenomenon eliminates the need for catalyst recovery steps-aligning with green chemistry concept. However, its catalytic performance can only achieved 61% conversion as reported. This followed by the high requirement for high-pressure equipment resulted in significant capital costs. As such, whilst the concept likely demonstrate strong environmental appeal, futther optimisation studies are required before it can be considered feasible for industrial application.

3.2 Key Concepts of Chemical Engineering and Process Implications

Beyond catalytic activity, the successful industrial scale of solketal production merely depends on overcoming key engineering hurdles, which are dressed in the reviewed literature.

3.2.1. Process Intensification Through Microwave Heating Assisted Method

Following the previous discussion on switchable CO_2 catalysis, how it can handle the synthesis process and can act as a functional catalyst, it is proven that chemical engineering solutions emerge not only from materials but also from energy input management. As Microwave used as a transformative approach, it offers a fundamentally different heating mechanism compared to conventional method of conduction or convection. Microwave has portion to interact with polar molecules in the medium which deposit energy volumetrically. This will accelerate collision of molecule and decrease the induction period.

For comparison, SAFACAM catalyst [20] only solidifies about 13,3% conversion despite the extended operation, an insufficient for industy standard. When exposed to the microwave heating, however, the conversion accelerates up to 98,1 % within 10 minutes of operation. The drastic rate of enchancement reduces reaction from hours to minutes. This means less requirement for such big reactor and open engineering opportunities for lower residence time, moreover the decreased energy consumptions. In the sense, process intensification of this approach leads to redefinine the scale up pathway, even though the practical integrations into large reactor remain as unaswered challenge.

3.2.2. Catalyst Stability and Deactivation

It is a fact for some catalyst that display excellent activity in the first experiment but then degrade very fast. Reports listed in the literature already show that without attention to longevity, the cost of frequent regeneration or replacement will cancel any benefit from the high initial conversion. In industry, the real concern is not one good data point, but rather the cumulative performance across many repeated cycles, where the operating condition often harsher than laboratory test. The deactivation of a catalyst may deliver two distinct pathways, namely, leaching-based and coking-based deactivation.

Though coking-based deactivation is less detailed in the literature, it is arised from incomplete substrate conversion and polymerisation of oligomeric intermediates that block active sites and pore structure. Carbon-based catalysts show particular vulnerability to coking mechanism due to accumulation of refractory carbonaceous residues on the catalyst surface and within the pore network. Extended reaction times and elevated temperatures exacerbate coking, explaining why thermal management represents a critical consideration for industrial-scale operations.

Leaching-based deactivation, meanwhile, involves dissolution and migration of active catalytic species from the solid matrix into the liquid reaction medium. For example, acid-functionalised carbon black derived from waste tyres (rCB) [19]. At the first cycle the catalyst achieve high conversion, nearly quantitative, suggesting promising activity. But after only one reuse, the performance drop below 1%, basically complete deactivation. This dramatic fall was likely because the sulfonic acid groups were not well anchored, and leaching occurs under liquid-phase reaction conditions. Such problem reveals how apparent activity in surface-rich carbon materials is often unstable, especially when the functional groups are too weakly bonded. A proposed remedy was to pre-treat the carbon with glucose before sulfonation, so that the active sites are more embedded. Following the modification, rCB increases resilience and sustains a glycerol conversion at 69% in the second cycle considered as promising reusable catalyst. The sulfated mixed oxides (SZZ), zeolitic WO_3/SAPO-34, acid-activated metakaolin clay, and also SAFACAM each could be reused for at least five cycles with relatively minor decline. However, none of these can match the TTPT ionic liquid performance in durability. TTPT takes dominance to the other reusable catalyst option up to 10 cycles. This phenomenon underlines that different catalyst families face distinct degradations e.g. leaching, pore blockage, or even structural collapse. Thus, material selections have to consider aspects that include crystallinity, defect tolerance, and hydrothermal stress.

3.2.3. Circular Economy Implementation

In theory, the circular economy focuses not only on reuse but also on creating new products that can be utilised by other industries or consumers. Some approaches focus on converting abundant waste into another valuable product for markets[4].

In this case of glycerol synthesis, Indonesia has to develop the key to robust and low cost catalysts. For instance, Acid-activated Malaysian kaolin was able to maintain a stable 73% glycerol conversion at 50°C even when crude glycerol was used, demonstrating of their practicality for tropical countries including Indonesia and other SEA countries.

This conditions is in cotrast to highly sensitive materials like the MOFs i.e Basolite F300, whose performance drops drastically from 84% with pure glycerol to only 14.8% when exposed to impure feedstock. Despite progress in catalyst design, stability remains unanswered. Most carbon catalysts, such as rCB show reduced longevity due to the formation of oligomeric byproducts. From this phenomenon, further research to enhance durability for industrial applications is needed. Promising solutions, such as carbon materials' pretreatment aimed to increase active sites. This should be explored to prevent deactivation of catalysts.

Another direction was to underscore the feasibility of process intensification. Using microwave heating instead of conventional methods might be the key to unlocking the higher conversion value and shortening the time of reaction. This also considerably suggests the potential for smaller, more efficient modular plants with less energy consumption. Ultimately, achieving true circularity requires the holistic integration of material science and innovative process engineering.

3.3. Future Research Recommendations For Indonesian Implementation

Whilst this review indentifies significant technological advances in solketal synthesis, substantial research gaps require addressing to provide practical and economical viability implementation within the Indonesian industrial context:

3.3.1. Priority Research Directions

Catalyst Development for Feedstock Robustness

Future research must prioritise catalysts tolerance for crude glycerol impurities without substantial performance degradation. Current laboratory catalysts assume pure feedstocks unsuitable for cost-effective industrial operation in the Indonesian context. Development of hybrid catalyst systems combining high activity with intrinsic impurity tolerance warrants systematic investigation.

Long-Term Stability Assessment Under Industrial Conditions

Extended durability testing (≥50 cycles) under industrially representative conditions, elevated temperatures, recycled feedstocks containing accumulated byproducts, variable humidity characteristic of tropical climates, remains absent from the literature. This represents a critical gap requiring systematic investigation to validate industrial scalability.

Enhanced Mechanistic Understanding of Deactivation

A detailed kinetic and mechanistic studies and deactivation pathways under varied operational conditions remain insufficient. Enhanced mechanistic understanding would guide rational catalyst redesign and mitigation strategies.

Microwave Technology Integration at Industrial Scale

Current microwave-assisted synthesis remains at laboratory scale. Design methodologies for integrating microwave heating into large scale continuous reactors, including comprehensive energy efficiency analysis and capital cost estimation.

3.3.2. Indonesia Implementation Gaps.

Comprehensive Characterisation of Indonesian Crude Glycerol

Systematic chemical characterisation of crude glycerol feedstocks by Indonesian biodiesel producers (varying by production facility, feedstock source and processing methods) requires further investigation. Current literature focused on generic crude glycerol specifications insufficient for process design and optimisation within the Indonesian industrial context.

Development of Catalysts Utilising Indonesian Mineral Resources

Investigation of clay and zeolite catalyst development specifically targeting minerals abundant in Indonesia. Thus, particularly clays from Banten, Sumatera, and other productive regions should require prioritisation. Utilising domestic geological resources substantially reduces import dependence, decreases production costs, and supports local value-added manufacturing.

Techno-Economic Feasibility Analysis

Detailed techno-economic analysis comparing competing catalyst systems and process configurations under authentic Indonesian operating conditions (labour costs, energy prices, waste disposal regulations, infrastructure constraints) remains absent. Such comprehensive analysis is needed for industrial investment decisions and technology transfer.

Environmental Impact and Lifecycle Assessment

Comparative lifecycle assessments evaluating environmental implications of competing catalyst systems and process configurations specific to the Indonesian industrial context require development. Such analysis is essential for policy alignment with national circular economy objectives and environmental sustainability targets.

4. Conclusion

This review concludes that solketal synthesis may be applied to different types of catalyst material with their own benefits and drawbacks. While resin catalysts can still be used as a benchmark, MOFs-based and zeolites also offer high activity but are prone to the presence of crude impurities. Simpler material, an acid-activated clays can be proposed due to their practical advantages and impurity tolerance which can reduce overall feedstock cost. To note, a simple extra steps of process intensification through microwave-assisted heating also improve glycerol conversion efficacy and energy utilisation. These ideas, if integrated properly, will likely contribute to sustainable chemical engineering solution for laboratory and industrial scale. By engineering waste to a valuable product like solketal supports the circular economy framework as proposed by the Ministry of Industry in Indonesia.

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