

Shaping Effective Practices for Incorporating Sustainability Assessment in Manuscripts Submitted to ACS Sustainable Chemistry & Engineering: Catalysis and Catalytic Processes

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The principles of green chemistry and green engineering provide guidelines for choosing reagents, catalysts, and operating conditions aimed at minimizing adverse effects of catalysts and catalytic processes on human health and the environment. The use of these *qualitative* principles in assessing the sustainability of catalysts and catalytic process concepts remains valuable. However, the editors of *ACS Sustainable Chemistry & Engineering* (ACS SCE) expect greater implementation of sustainability metrics to quantify the extent to which the adverse effects of incumbent catalytic technologies are mitigated by the innovations described in manuscripts submitted to the journal. Authors are strongly encouraged to adopt these quantitative analyses not only to identify and quantify the contributions to sustainability of innovations reported in manuscripts but to also reveal economic and environmental problem areas that must still be addressed in future research to achieve a sustainable process.¹ The outcomes of such analyses also inform investments in research that will eventually lead to the commercialization of green chemical technologies. This editorial suggests guidelines for incorporating quantitative sustainability assessments in manuscripts describing catalytic processes. We have classified the guidelines based on the types of manuscripts we receive in the area of catalysis. Please note that guidelines for electrocatalysis will be provided in a separate editorial on sustainability metrics for electrochemical processes and will appear later this year.

■ GENERAL CONCEPTS

Catalysis, by improving reaction rates and selectivities, contributes to resource-efficient chemistry and, therefore, inherently promotes sustainability.² Beyond providing conversion, yield, and selectivity values, we encourage authors to use metrics, such as turnover number (TON) and turnover frequency (TOF), to characterize the intrinsic activity of catalysts. Such metrics aid in reliably comparing the intrinsic performance of the new catalysts against incumbent catalysts. Such metrics, along with information on a catalyst's durability, provide valuable information about the sustainability of the process.³

When reporting on catalyst recyclability, we encourage authors to report catalyst behavior in either multiple repeated cycles in a batch reactor or time-on-stream stability test in a continuous flow reactor. Such studies are essential for establishing two key parameters in a good catalyst: (1) How

stable is its activity? (2) How long can it function? In particular, it is good practice to evaluate the performance of recycled catalysts at less than an equilibrium conversion (e.g., <50% of equilibrium conversion), in order to reliably discern either stable or decreasing activity in subsequent cycles.⁴ Better yet is the analysis of initial rates obtained from kinetic plots,⁵ which can reveal either induction period effects or rapid initial loss of activity.⁶ Additionally, reporting the maximum TON value for a catalyst is a good metric for intrinsic catalyst robustness and should be implemented more systematically.⁷ Importantly, authors should report in detail the procedure for each cycle, such as any catalyst washing or treatment involved.

A metric used in catalyst recycling studies to report catalyst stability is the extent of catalyst metal leaching into the reaction mixture. This is often quantified using elemental analysis methods, such as inductively coupled plasma atomic emission spectroscopy (ICP AES).⁸ In any metal-based catalysis, it is also important to test the amount of metal residue present in the end product. Care should be taken in selecting adequate digestion methods for metals, such as Ru, which is susceptible to volatilization in the form of RuO₄.⁹

Many catalytic processes involve the use of auxiliary substances, such as water, organic solvents, and mineral acids and bases. In addition, they involve solvent- and energy-intensive separation and purification steps for recovering the product and catalyst. Considering that the mass of solvent is usually much greater than the mass of all other reagents, it is important for authors to discuss efforts in reducing the number and quantity of solvents used and in choosing greener solvents. The choice of greener solvents is usually based on its impact on the environment, health, and safety (EHS).¹⁰ The use of certain solvents (particularly chloroform, dichloromethane, and benzene) are subject to international regulations, banning their import and usage, such as Europe's Registration, Evaluation, Authorization and Restriction of Chemicals (REACH).¹¹ Similar, the choice of greener acids and bases can also reduce environmental impacts.¹² The choice of greener auxiliaries used in purification steps, such as desiccants,

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chromatographic column packings, brine, and acid/base washes, has not evolved in the same way as solvents. The overall recommendations to authors are to assess the use of auxiliary substances and to use either benign materials or avoid workups, wherever possible. For reporting the overall sustainability of auxiliary substances, the environmental impacts during their manufacture from raw materials must also be assessed using appropriate metrics. Some suggested approaches in this regard are provided in a following section on pharmaceuticals, where manufacture tends to be solvent intensive.

Manuscripts that either minimize or totally avoid the use of solvents in catalysis are welcomed. An emerging area of interest is catalysis via mechanochemistry, which has the potential to limit the quantity of solvent used,¹³ although systematic use of metrics is needed to confirm the sustainability benefits of such an approach.^{14,15} In the context of photochemistry, quantum yield is a metric that needs to be more systematically reported in order to evaluate the utilization of each photon and, ultimately, energy.^{4,16}

■ PHARMACEUTICALS

The molecular complexity of active pharmaceutical ingredients (APIs) requires multistep synthesis that consumes large amounts of reagents. The pharmaceutical industry is the most tightly regulated and generates significant waste per unit mass of product produced. Solvents are required for both upstream and downstream processes, and the choice of the reaction media involves a complicated decision process involving cost, safety, and sustainability.¹¹ In the past few decades, Pd catalysis has become enormously popular for API synthesis due to its versatility, robustness, and selectivity in a number of C–C and C–X bond-forming reactions. However, the use of precious metal catalysts is unsustainable.¹⁷ Efforts to replace Pd with nonprecious metals or earth-abundant catalysts have not been particularly successful, as the alternative catalysts often require much higher catalyst loadings and expensive ligands. This is particularly important as the presence of metal residues in APIs is also tightly regulated;¹⁸ their removal and recovery often require complex workup procedures that are deleterious for the sustainability of the process. The sustainability aspects underlying the catalyst metal choice and preparation methods are discussed in more detail in a separate section below.

The CHEM21 toolkit¹⁹ was developed in 2015 by a consortium of academics, pharmaceutical manufacturers, and small and medium enterprises (SMEs) as a means of evaluating the sustainability of reactions. The tool encompasses a range of well-established metrics, including atom efficiency (AE) and process mass intensity (PMI), along with three new metrics: overall efficiency (OE), renewable percentage (RP), and waste percentage (WP), as well as safety and environmental assessment of solvents. The toolkit is free to download (as an Excel file) and easy to implement in upstream and downstream processes at different scales. Authors submitting manuscripts in catalysis are strongly encouraged to utilize this free resource to evaluate the sustainability of their catalytic methodology. For researchers engaged in the development of early, late, or commercial processes, a green manufacturing metric, known as the innovation green aspiration level (iGAL), was developed by the ACS Green Chemistry Institute and a consortium of pharmaceutical manufacturers.²⁰ Using the molecular weight of the active pharmaceutical ingredient

(API) as an indicator of its complexity, with two other inputs (number of constructive steps and complete E-factor, cEF), a free-to-access online scorecard²¹ calculates the impact of implementing green process inventiveness and improvements and delivers an attractive colorful graphic output that compares the relative process greenness (RPG) between processes, as well as the industry average, as defined by improvements to ideality and complexity, as well as overall waste reduction.

■ BIOCATALYSIS

Driven by the need for more sustainable chemical processes, enzymatic catalysis is increasingly used not only in the pharmaceutical sector but also in the fine and specialty chemicals sector. Enzymatic reactions developed at the research laboratory scale often showcase excellent sustainability indicators.²² It is important to quantitatively confirm this for new biocatalytic processes by reporting selectivity, specificity, and specific activity. Although activity-based specifications are widely used to express the enzymatic activity (U or U mL^{−1}), mass-based or mol-based specific activity (mol g_{enzyme}^{−1} s^{−1} or mol mol_{enzyme}^{−1} s^{−1}) is useful and should also be provided. This is particularly useful, for example, when the enzyme is supported on a carrier to enhance recoverability, recyclability, and stability; it is essential to quantify the impacts of such procedures on their intrinsic activities (residual activities).

Moving further, economic metrics, such as volumetric productivity, are needed to assess the potential of the process, as this will ultimately drive the transition to commercialization. Often, biocatalytic reactions are performed in water, in rather dilute conditions. Although water is often bluntly claimed to be the greenest solvent, it is crucial to acknowledge that water decontamination and isolation of the products from water can be complicated.²³ Any required treatment of water (separation, purification) will negatively impact the competitiveness of the process. We suggest to systematically include the calculation of the complete E-factor (cEF, which includes water) or to report a solvent intensity metric (kg solvent used per kg of product) to help position the new process with respect to existing technologies. In this way, biocatalytic processes that can be operated at higher substrate/product concentrations will be positively differentiated.

Finally, many enzymes require the use of cofactors for which separation and reuse are often complicated, and this must be considered in the sustainability assessment discussion. We encourage contributors to demonstrate efficient strategies for cofactor regeneration, coimmobilization, or recycling.²⁴

■ CATALYST PREPARATION

Whether homogeneous, heterogeneous, or enzymatic, the production of the catalyst itself may impose an environmental burden that should be considered in our efforts to transition toward more sustainable chemical processes. For example, in the production of biofuels, greenhouse gas emissions associated with catalyst production have been reported to range from less than 1% to greater than 10% of cradle-to-gate greenhouse gas emissions for biofuels,²⁵ depending on the catalyst type and loading. Significant impacts associated with catalyst preparation may be more common than is widely recognized. Metal complexes are often prepared through multistep procedures with adverse sustainability metrics. Although enzymes are considered as renewable and biode-

gradable catalysts, their production and purification steps must be considered for reliable sustainability assessment of bioprocesses.²⁶ In addition to the biologic origin of the enzyme, and possibly its engineering, details should be provided on its expression and on its purification. It is worth noting that the manufacture of widely used heterogeneous catalysts, such as TiO₂, imposes environmental burdens during the mining of the raw material as well as during processing that requires the use of large amounts of sulfuric acid.

Manuscripts focusing on the development of greener manufacturing of catalysts are welcome. In this case, metrics associated with catalyst production should be used to demonstrate the superiority of the alternative method. Yet, improvements in catalyst production methods should not come at the expense of inferior catalyst performance. Any claim of a greener catalyst preparation method should be accompanied by clear evidence that its performance remains at least at the same level as the catalyst being replaced. Ideally, both the catalyst preparation step as well the catalytic process should possess favorable metrics for the overall process sustainability. However, a new catalyst should not be summarily rejected because its preparation requires, for example, a slightly higher calcination temperature or the use of an additional solvent. It can still represent a viable option if such drawbacks in catalyst preparation are largely outweighed by process improvements resulting in more durable catalyst performance at a larger scale. We welcome discussions on such nuanced aspects.

■ CATALYSIS WITH EMERGING FEEDSTOCKS (BIOMASS, CO₂, RECYCLED CARBON SOURCES)

The utilization of renewable and recycled carbon sources opens up new opportunities toward a more sustainable chemical industry. However, the replacement of fossil resources to produce chemicals is not an automatic guarantee of sustainability. Instead, a quantitative measure of the net CO₂ footprint of the feedstock during cultivation, harvesting, and transportation must be accounted for. The estimation of the carbon footprint of biomass is commonly practiced today,²⁷ with a lower and more negative carbon footprint connoting favorable sustainability of the manufacturing process. For catalytic reductions, the use of renewable hydrogen in wood processing and sustainable forest management are options to consider. Rigorous CO₂ footprint analysis for emerging feedstocks, such as plastic waste and CO₂ itself as recycled carbon, may be difficult with data available today, presumably because these fields are less mature. However, as more data on these emerging feedstocks become available, such analysis should become possible. The process to recycle waste is often material and energy intensive. The adverse environmental effects created by such processes should not nullify the environmental advantages of recycling. A waste recycling strategy, in general, must therefore address this issue clearly.

■ LIFE CYCLE ASSESSMENT APPROACHES FOR CATALYTIC PROCESSES

One cannot meaningfully compare conventional and alternative processes without reliable data. The data for conventional manufacturing processes are usually sourced from the open literature, including patents. Simulations of greener alternatives must rely on reported laboratory-scale data to develop detailed process flow diagrams that include down-

stream separation of products, recycling of unreacted feedstocks and catalysts, and safety considerations. Detailed process engineering simulations like these form the basis of technoeconomic and environmental impact analyses. Rules of thumb for process engineering attributes that promote sustainability are particularly encouraged. Examples include relative volatilities of reactants and solvents to reduce energy intensity, the extent of solvent usage relative to feed/product concentrations, and reactor operating temperatures for effective heat integration. Cradle-to-entrance gate and gate-to-gate impact assessments delineate the environmental and health burdens associated with raw material extraction and the chemical manufacturing process. Such an approach thus encourages a life-cycle approach to identifying bottlenecks and recommending process improvements for enhancing sustainability. Publicly available data, reported by chemical companies and power generating stations, serve to compare the predicted environmental impacts (emissions) from various unit operations at manufacturing sites.

LCA tools, such as SimaPro,²⁸ GaBi,²⁹ and GREET,³⁰ have been employed to benchmark alternative process concepts with conventional technologies. Process LCA methods assess resource inputs from gate-to-gate based on the simulated process.³¹ For performing cradle-to-gate LCA, the needed upstream information on the environmental impacts of making the raw materials is usually obtained from the referenced databases. However, such information may not always be available.³² In such cases, the economic input–output LCA (EIO-LCA) method based on economic activities and their environmental impacts may be useful.^{33,34} A hybrid LCA method that combines the advantages of process LCA and EIO-LCA methods has also been employed and helps further delineate the upstream impacts from the gate-to-gate impacts.³⁵ Readers may find a review and application of the hybrid LCA methodology for comparing two biomass-based p-xylene productions useful.³⁶

It should be recognized that chemical process design technoeconomic analysis (TEA) and LCA are usually performed at a low technology readiness level (TRL) based on the prevalent efficiencies of the unit operations, such as process yields and separations. Prospective LCA builds on current LCA projections to project future process and product improvement scenarios,³⁷ thereby providing a trajectory for current technologies to improve sustainability attributes. As an example, upstream environmental burdens stemming from the use of fossil-based feedstocks and energy sources are dominant in a majority of the current technologies, even though their gate-to-gate profiles may be far superior to incumbent technologies.³⁸ The prospective LCA approach can thus provide a trajectory of environmental impact changes over time when renewable carbon and energy sources become increasingly available to make chemical products.

Beyond the process, the disposal, management, and recycling of the waste products as a carbon source are receiving increased attention in the context of promoting a circular economy. As noted in an earlier section, sustainability metrics associated with waste recycling are garnering attention only recently and are of interest to ACS SCE.

In summary, we hope that these guidelines are useful and encourage authors to incorporate sustainability metrics and LCA tools as appropriate in their research. We believe that they will enhance the impact of their contributions, and we look forward to receiving such manuscripts.

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