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A Study on Chemical Reactor Concepts for Green Chemical Processes

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ABSTRACT

Green synthesis of chemicals has a significant positive impact on environment. Because of the impact on pollution, energy consumption, air quality, and global warming, the scientific community and chemical industry are becoming increasingly interested in concepts like atom economy; renewables feed stocks and sustainable solvents as an inseparable part of green synthesis. For example, solvent losses are a major source of organic pollution, and solvent cleanup consumes a lot of energy and more environmentally friendly or sustainable solvents have been proposed and developed over the last three decades. Green chemistry for chemical synthesis addresses future challenges in chemical synthesis by developing new synthetic schemes and apparatus to simplify chemical production, developing novel reactions to maximize desired products while minimizing by products, and seeking ecologically and environmentally safe solvents.

Keywords: Chemical reactor; Chemical engineering; Green chemical processes; Atom economy; Organic pollution

INTRODUCTION

Chemical engineering is concerned with the transition of chemicals from one form (raw materials, including renewable and non-renewable resources) to another (products) that is beneficial to mankind and meets the needs of a comfortable life [1]. This process of transforming raw materials into finished goods invariably results in the production of undesirable byproducts (pollutants) and the depletion of renewable and non-renewable resources. The demand for useful chemical goods (such as cement, sugar, pulp and paper, pharmaceuticals, petroleum, and petrochemicals, among others) is constantly increasing as the world's population and living standards rise as a result of economic growth [2]. Chemical production capacity in Asia alone, the world's most populous area, is predicted to expand by four to six times its capacity in 2000, by 2025. This would result in a faster use of natural resources such as fossil fuels and fresh water, as well as a steeper rise in pollution levels in the environment. If left uncontrolled, the long-term and short term environmental consequences could jeopardize the very existence of life on earth. Thus, it is extremely important that we assign to each product an environmental factor, or e-factor, which is defined as the amount of trash generated per unit mass of a product produced and, as such, is a measure of the environmental impact of the different chemicals produced in the industry [3]. Contrary to popular belief, chemical industries producing high value added chemical products such as pharmaceuticals and chemicals contribute more to environmental degradation than refining and bulk chemical industries. Economic growth and development at the expense of environmental degradation is unsustainable in the long run [4]. The need and the necessity for striking a suitable balance between economic development and environmental degradation have

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given birth to the concept of sustainable development [5]. In 1987, the UN world commission on environment and development said that the definition of sustainable development is, meeting the needs of the present without putting the ability of future generations to meet their own needs at risk. A sustainable process or product is one in which resource consumption and waste generation are kept at acceptable levels while making a positive contribution to societal needs and generating long term product to the business enterprise. Thus, sustainable development involves complex interaction between industry, society, and the ecosystem [6]. Although chemical industries contribute to 12–15% of the Gross Domestic Product (GDP) of any developing nation, they are generally perceived by society as a major source of pollution and a major cause for environmental degradation. So, there is a greater expectation on the chemical engineering community to play a responsible role in protecting the environment by way of developing and adapting newer technologies and newer methods of production that are friendlier to the environment. Any technology that is environment friendly is known as 'green technology'. Green technologies are sustainable technologies of higher material and energy efficiencies that use renewable resources and prevent or minimize pollution at the source rather than treating it at the end of the pipe [7]. One may consider the total pollution generated in producing a chemical as a product of total global population, consumption per capita and the inefficiency of the process. It is not possible to regulate or reduce the population growth or consumption per capita as any attempt to curb these factors through governmental regulations would lead to social and political unrest and economic instability. So, the only viable option for reduction or minimization of pollution is to develop newer technologies and processes that are more efficient in terms of material and energy utilization and minimization of waste generated [8].

MATERIALS AND METHODS

Principles of Green Chemistry and Green Chemical Engineering

In their 1998 book, Paul Anastasia and John Warner developed the concept of green chemistry as a philosophy for the design of chemical products and processes that decrease or eliminate the use and creation of hazardous compounds [9]. The founders of green chemistry had established 12 fundamental principles, the application of which in the practice of chemical engineering is intended to result in the development of environmentally friendly products and processes. The following are the 12 fundamental principles of green chemistry [10].

- **Prevent waste:** Design the process such that waste is prevented at the outset rather than treating or cleaning up waste after it has been generated.
- **Maximise atom economy:** Synthesise or design methods that maximise incorporation of all material used in the process into the final product so that the generation of waste is minimised. Select an alternative synthesis route or raw material such that the amount of side reactions generating undesired by-products is minimal. Atom economy is taken as a measure of how efficiently the raw material is used. Raw materials yielding maximum atom efficiency are selected.
- **Design less hazardous chemical synthesis:** Synthesise or design methods that use and generate substances that minimise toxicity to the environment.
- **Design safer chemicals and products:** Design chemical products with least toxic contamination to the environment.
- **Use safer solvents and auxiliaries:** Reduce the use of solvents that cause pollution. If necessary, use solvents that are benign to the environment. In this context, supercritical CO_2 ($scCO_2$) and ionic liquids show remarkable potential as eco-friendly substitutes for solvents that are toxic to the environment.
- **Design for energy efficiency:** Design processes for higher energy efficiency so that net consumption of energy (fuel) is minimised and the impact of energy usage on the environment is reduced. The processes that operate at ambient temperature and pressure consume less energy and are preferred over high-temperature and high pressure systems. The possibility of reducing energy and material consumption through appropriate processes and heat integration should be explored. Novel combo systems that integrate reactors with mass exchangers offer plenty of opportunities for the design of energy-efficient chemical processes.
- **Use renewable feed stocks:** Choose a raw material or a feed stock that is renewable rather than depleting, wherever possible. The alternate route for synthesis of chemicals using biomass as feed stock is an option to be explored. For example, biochemical synthesis of adipic acid using glucose as a feed stock is an eco-friendly alternative to the traditional chemical synthesis of adipic acid using benzene as feed stock.
- **Reduce or avoid the use of chemical derivatives:** The use of chemical derivatives should be avoided or minimised as this can lead to the need for additional reagents and further generation of waste.
- Catalysis can be used in place of stoichiometric reagents. When compared to synthesis approaches that use stoichiometric reagents, catalyst based synthesis produces less pollution. As a result, a catalyst is critical in the design of ecologically friendly chemical processes. With the right choice and design of solid catalysts, waste reduction can be made much better.
- Chemical products should be developed so that when they no longer serve their purpose, they disintegrate into benign byproducts and do not persist in the environment. For example, biopolymers such as Poly Lactic Acid (PLA) and Polyhydroxyalkanoates (PHA), used as substitutes for chemical plastics, exhibit excellent biodegradability, unlike plastics.

- **Real time analysis for pollution prevention:** Create analytical procedures that enable real-time monitoring and control before dangerous substances form.
- **Minimise potential for accident through safer chemistry:** Substances used in chemical processes should be chosen to minimise the potential for chemical accidents including releases, explosions, etc.

Chemical Reactor

Any chemical plant can be perceived as a system of units arranged in a particular sequence of material processing steps required to transform raw material to a desired product. All processing units in a chemical industry can be broadly grouped into three sections: raw material pretreatment section, reactor section and separation or purification section of these three sections, the reactor section in which key chemical transformations take place is the heart of chemical processing and any improvement in the performance of the reactor section is likely to have a major impact on pollution prevention [11]. Thus, chemical reaction engineering plays a central role in green chemical processing. Although the principles of green chemistry provide a road map to the development of green processes, it is the selection, design and operation of the reactor that determines whether a process would be successful or not. In most chemical processes, the choice of the reactor and its operation has a very strong influence on the number and type of separation units required on the upstream and downstream sides and hence has a profound impact on the environment. Chemical reaction engineering provides the methodology for quantifying the reactor performance as a function of design and operational variables. Reactor performance, which is measured in terms of fractional conversion of reactants and product selectivity, is influenced by several factors such as feed rate, reactor size, temperature, kinetic rate, transport rate, mixing and flow patterns [12]. Appropriate quantification (or modeling) of reactor performance requires a multi scale approach involving system characterization in a wide range of scales, from molecular to macro. So, pollution prevention through proper choice and operation of the reactor requires the related issues to be addressed in all these scales. At the molecular level, understanding process chemistry helps to achieve maximum atom efficiency, understanding complex reaction mechanisms leads to the development of appropriate kinetic rate expressions used in reactor design and understanding the mechanism of catalytic reactions leads to the design of a catalyst for achieving maximum selectivity [13]. At a micro scale, understanding fluid mixing and transport in eddies, transport in multiphase systems, transport within pores of a catalyst pellet and the effect of local transport on reaction rate is crucial for the design of reactors that achieve optimum performance. At a macro scale, understanding the effect of hydrodynamics on reactor performance is crucial for scale up and operation of reactors. Thus, a multistate reactor modeling approach, which integrates the system description at all these three scales (molecular, meso and macro), is needed for the design of reactors to achieve optimum performance. Multistage reactor modeling and design approach requires highly sophisticated and advanced computational tools and experimental techniques. A proper quantification of reactor performance requires an appropriate description of how the reacting species are brought into contact by fluid mixing. In a multiphase reactor, one should be able to describe the flow and mixing patterns in each one of the phases. In the traditional approach to reactor design, either plug low or completely mixed low is usually assumed in each one of the phases [14]. If this assumption does not match with the experimental observation, an axial dispersion model is assumed, and the model parameter (peclet number) tuned to match the experimental measurements. However, this traditional approach lacks capacity to describe systems having complex fluid mixing patterns. A better description of the low, mixing and phase contacting pattern is required to develop more realistic reactor models. In this context, sophisticated Computational Fluid Dynamics (CFD) based models can capture and describe the complexities of fluid mixing more realistically. Thus, CFD is an important computational tool useful for the design of novel reactors. However, sophisticated experimental measurement techniques are necessary to get information on fluid velocity and turbulence to validate the CFD based models [15]. The selection and design of a catalyst play a crucial role in achieving high reactor performance and making the process environmentally benign. On selecting a catalyst that is appropriate for a particular application, the catalyst can be tailor made for optimum yield and selectivity. Advanced experimental and computer simulation techniques are used to study the surface topology, adsorption characteristics, pore structure and transport properties that are useful for the design of a catalyst having requisite characteristics. A novel catalyst design combined with advanced reactor design technology can make any process economically viable and environmentally beneficial.

RESULTS

Addressing challenges by devising innovative and fundamentally novel chemistry throughout the synthetic processes.

Chemical feedstocks: Currently, the primary feedstock for chemical products is nonrenewable petroleum, which is quickly depleting for both chemical and energy demands. But nature provides a lot of renewable biomass in the form of carbohydrates, amino acids, and triglycerides that can be used to make organic products. However, a big problem with using renewable biomass as feedstock is that it needs new chemistry to change large amounts of biomass in its natural state, without a lot of functionalization or protection.

Reactions: Reactions play the most fundamental role in synthesis. The green chemistry idea is to find new chemical reaction pathways and reaction conditions that could make chemical synthesis better in terms of using less energy and resources, making better products, making the process easier, and keeping people and the environment safe.

Solvents: Solvents are utilized as auxiliary substances in chemical synthesis. They are not intrinsic elements of the reacting compounds, yet they play a crucial function in chemical production and synthesis. By far, the largest proportion of "auxiliary waste" is related to solvent usage in the majority of chemical operations. In a conventional chemical process, solvents are widely employed for dissolving reactants, extracting and washing products, separating mixtures, cleaning reaction apparatus, and dispersing products for practical uses. Although the advent of numerous novel organic solvents resulted in tremendous improvements in chemistry, the legacy of these solvents has resulted in numerous environmental and health hazards. As a result, as part of green chemistry initiatives, a variety of cleaner solvents have been examined as alternatives. In classical chemical synthesis, the major function of solvents is to allow mass transfer in order to control chemical reactions in terms of reaction rate, yields, conversions, and selectivity. This is accomplished by dissolving the reactants in diluted homogenous mixtures. After the reaction, the final product must be taken back out of the solvent using methods that take a lot of energy (**Figure 1**).

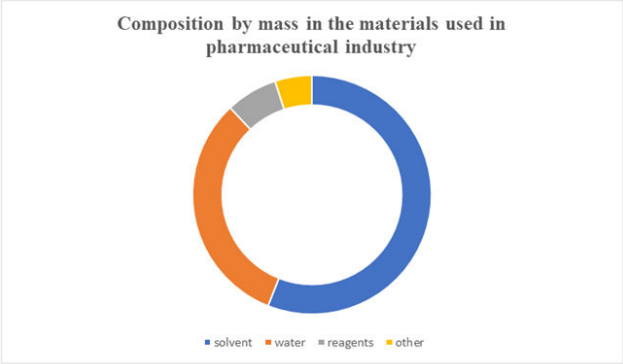

Figure 1: Composition by mass in the materials used in pharmaceutical industry.

Solvents are typically the determining factors in assessing the environmental impact, cost, and safety of a process. Process solvents can account for 80-90 percent of the non-aqueous bulk of API manufacturing materials. Other reports have indicated that solvents account for fifty percent of pharmaceutical manufacturing's greenhouse gas emissions. According to GSK, less than half of spent solvents are recycled or repurposed.

Atom economy: Conventionally, yield maximization and product selectivity are the driving forces behind chemical synthesis. Little concern was given to the use of numerous reagents in stoichiometric amounts, which were frequently not absorbed into the target molecule and would result in the formation of substantial side products. In a balanced chemical reaction, on the other hand, a simple addition or cycloaddition involves all of the atoms that were in the starting materials. Recognizing this fundamental phenomenon, the atom economy was introduced in 1991 as a set of coherent guiding principles for evaluating the efficiency of specific chemical processes. These principles have since been incorporated into the "twelve principles of green chemistry" and have altered the way in which many chemists design and plan syntheses. An atomic economy aims to maximize the assimilation of starting components into a reaction's ultimate result. The second implication is that, if maximum integration cannot be accomplished, the quantities of side products should ideally be negligible and harmless to the environment. The reaction yield is solely concerned with the amount of the desired product that is isolated relative to its theoretical amount. Atomic economics takes into account all used reagents and undesired byproducts alongside the desired result. Substitutions and eliminations, for example, are uneconomical classical reactions with inherent wastes that cannot be avoided. The intended modes of reactivity are simple additions, cycloadditions, and rearrangements. Reaction Mass Efficiency (RME) and Mass Intensity (MI) are supplementary concepts used to assess the efficiency of synthetic reactions in consideration of the reaction yield. For example, isomerization of propargyl alcohols into conjugated carbonyl compounds is an atom efficient method for the synthesis of conjugated carbonyl compounds. By replacing the usual two steps stoichiometric reduction and oxidation sequence with a ruthenium catalyzed redox isomerization of propargyl alcohols into enones, a catalytic enantioselective complete synthesis of acetylenes can be made that is both efficient and selective for the right kind of atom.

Biocatalysts: Using biocatalysts to aid chemical reactions is one of the most environmentally benign ways of production. Through millions of years of evolution and "sustainability," nature has built highly efficient and selective transformation mechanisms. The potential utility of numerous natural catalysts, such as enzymes, entire cells, and catalytic antibodies, for chemical synthesis is becoming increasingly apparent. Biocatalysts frequently results in reaction speeds and selectivity, such as enantioselectivity, that surpass the capabilities of chemical catalysts. These advancements have endowed the synthetic chemist with powerful and parallel tools. However, the high substrate specificity of enzymes poses a problem for synthetic chemistry, which requires broad substrate applicability. The new and intriguing development of "directed evolution" offers the possibility of using biological catalysts to address this problem.

Extraction/Separation: Traditional manufacturing scale continuous separations are among the most energy intensive unit activities, with distillation contributing the most. The implementation of less energy intensive small scale continuous separations could provide a "green" niche for continuous manufacturing, not only for energy conservation but also for waste reduction. There was a successful demonstration of the automation and deployment of small scale gravity based liquid/liquid separations. Typical monitoring utilizes either the capacitance differential between the aqueous and organic phases to trigger the draining of the bottom phase by opening a valve, or a web camera and a reference object at the interface of the two liquids. Even though this method is elegant, it needs to be calibrated for each response stream and has to start slowly because of settling times and dead volumes. Due to the predominance of surface forces at this scale, they offer

alternatives for performing efficient extractions. In typical extraction techniques, de emulsification is a concern since emulsions can be resilient and remain for extended periods of time. Utilizing this predominance of surface forces in micro channels resembling plates for the fast de emulsification of two liquids, use of capillary separators with a wide variety of surfaces and wetted materials has been permitted. Due to their selective wettability qualities, chemically inert membranes, such as Polytetrafluoroethylene (PTFE), can be utilized to selectively extract, separate, and isolate the target API during or after a synthesis. They are utilized extensively in multi-step reaction sequences. Once adjusted, these technologies provide a genuinely continuous technique to complement the continuous production of the active pharmaceutical ingredient. Membrane based phase separation is a robust and green unit operation that shifts to a fully continuous methodology for the purification and separation of API reaction streams. Membrane based phase separation's minimal energy requirements and use of inexpensive and disposable materials make it a green unit operation. An important type of membrane based separation is pervaporation that has not been widely used in the pharmaceutical industry, in part because it is less commercially mature than more conventional separation techniques such as distillation. Recovery of organic solvents (such as isopropanol) from industrial waste streams or continuous processing of commodity chemicals, such as ethanol, has proved the commercial viability of evaporation. Evaporation may also be used to dehydrate API streams in a multi step, continuous process. Numerous chemical processes, such as Grignard or litigation, might be harmed by excessive water levels. Therefore, researchers have few in line options if they desire to incorporate a moisture sensitive reagent at a late stage of a telescoped synthesis. Evaporation is an interesting option for dehydrating water sensitive process streams due to its ability to shatter solvent zoetrope's. Evaporation applications are constrained by the chemical compatibility of contemporary evaporation membranes. Multiple successive units or recycling loops are frequent in evaporation systems to provide sufficient moisture removal, but they increase processing time and expense. When the composition of the reaction stream permits, a form of evaporation that employs vapor permeation can be used. A hybrid vapor permeation distillation unit can help dehydrate flow streams by feeding steam from the distillation unit to the vapor permeation unit.

In line concentration: The difficulty of cumulative dilution is intrinsic to the design of telescoped multi step syntheses. Simply beginning with highly concentrated reagent streams is the most obvious, although not necessarily optimal, option. At high concentrations, solubility becomes an issue, as does the stress on pumps that may be required to run undiluted chemicals in order to achieve the appropriate concentration downstream. As previously stated, rotary evaporators can be utilized in a semi continuous manner to improve stream concentration. Tiny evaporators usually have poor chemical compatibility, low flow rates, and limited solvent stream composition variation created a prototype in line evaporator that successfully decouples the rate of solvent removal from its boiling point. The method could remove low to medium boiling point solvents (T 100 degrees Celsius), but high boiling point solvents such as water, DMF, and n-BuOH were difficult to remove. At 80 degrees Celsius, the elimination of the aforementioned solvents was 32%, 56%, and 86%, respectively. Using this prototype (MeOH/Toluene) and getting rid of excess nitro methane, a process reagent that would have stopped the subsequent Michael addition reaction, solvent switching was achieved.

DISCUSSION

Solids Handling

Certain chemical reactions necessitate the addition of solid reagents to a process stream, such as magnesium turnings for Grignard reagent. This is possible by employing a Continuous Stirred Tank Reactor (CSTR) and the slow addition of material to a continuous stream. It has been claimed that magnetically stirred slurries can be pumped, but particle settling and probable blockage pose a risk to the process. Solid addition could alternatively make use of mechanical feeders, such as hoppers or screw feeders, similar to equipment used for solid formulation. Solids generated during a reaction pose a problem for processing, since their progressive deposition on reactor walls can exacerbate pressure decreases and ultimately choke channels. During such occurrences, process safety and product quality issues develop. Low solubility particles in the process stream agglomerate and finally clog reactor tubes.

Process design (e.g., avoiding sharp corners, adding pressure pulses) and process parameter control assist in preventing clogging occurrences. The use of fluoropolymer tubing prevents the deposition of particles on reactor walls, and the replacement of tubing on a quarterly basis is standard operating procedure. In the pharmaceutical business, CSTRs have been exploited for continuous crystallization or for synthetic paths known to generate solids. Solicitors, such as haptic motors or ultrasonic probes, aid in preventing blockage. Researchers also have access to mechanically agitated mini reactors (Coflore), Multijet Oscillating Disk (MJOD) reactors, Continuous Oscillatory Baffled Reactor (COBR) reactors, and gas liquid segmented flow. However, no method has gained universal acceptance.

Crystallization

In an end to end process, the API must be extracted, purified, and transferred to the drug product phase. It is a crucial step in the production process, as the crystallization conditions determine the product's purity and physical properties, such as particle size and morphology. These material characteristics can have a substantial impact on subsequent processing and must thus be regulated during the crystallization phase. Crystallizations are one of the hardest unit processes to turn into a continuous process. This is because they are sensitive to things like temperature, mixing,

and residence time. As they are often the final step before formulation, it is vital to have a process that consistently achieves the specified purity and material characteristics, *i.e.,* avoids uncontrolled process performance and quality variation. Similar to other process steps, PAT monitoring can be relied on heavily to validate process control. Continuous crystallization can offer significant benefits in the form of reduced Capital Expenditure (CapEx) and Operational Expenditure (OpEx) as well as a more effective use of energy and resources (G6). Continuous crystallizations provide several potential benefits for API attributes:

- Elimination of batch-to-batch variation;
- Particle size regulation for more uniform slurry and powder qualities;
- Enhancement of drying, filtration, dissolving, and bulk flow properties
- Scalability of methods

In practical terms, Mixed Suspension Mixed Product Removal (MSMPR), COBR, CSTR, and Plug Flow Reactors (PFR) account for the majority of platforms now utilized for continuous crystallization. In addition to worries about fouling or encrustation of reactor walls, propellers, mixers, etc., chemical inertness and compatibility are crucial design factors. Examples of continuous reconfigurable crystallization processes. Similar to its cousin in synthesis, PAT will be essential to the effective integration of continuous crystallizations down the line. At this point in the procedure, researchers have access to a variety of techniques, including FTIR, Near Infrared Spectroscopy (NIR), Focused Beam Reflectance Measurement (FBRM), and Particle Vision Measurement (PVM). A suite of PAT has the potential to be used on continuous crystallizers for monitoring of concentration, Particle Size Distribution (PSD), and crystal shape, which is essential for real-time process monitoring and control. Despite advancements in continuous crystallization platforms, the principles of crystallization (nucleation, growth, polymorphism, and crystal structure-property connections) remain poorly understood. Consequently, there is a significant difference between the advancements and new capabilities of flow chemistry and continuous crystallization. Similar to previous unit operations, basic research efforts must be increased to enable the full adoption of continuous production if one wants to successfully develop an integrated process train for drug substances.

CONCLUSION

This research promotes the minimal consumption of nonrenewable natural resources and the maximization of pollution prevention through the use of green chemical processes in a chemical reactor. This article emphasizes the significance of establishing environmentally friendly processes and incorporating green chemistry and reaction engineering into their design. In recent years, corporations, governments, and researchers have shown an increasing interest in "green processes" and "green products" as the concept of sustainability has become a focal point. A "green process" refers to the precise steps that reduce the environmental impact of a product's production processes.

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