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Photon Management in Photochemical Synthesis and Reactor Scale-Up

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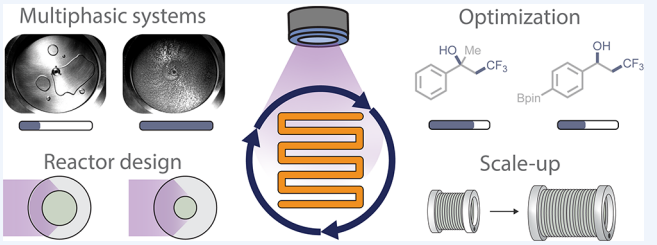
CONSPECTUS: Photochemical methods have become indispensable in modern organic synthesis by enabling unique reactivities under mild conditions through electron transfer, energy transfer, and other radical-based pathways. In contrast to thermally driven reactions, however, photochemical processes are fundamentally governed by the delivery and utilization of photons. Wavelength, light intensity, photon flux, optical path length, and reactor geometry collectively determine how efficiently photons are absorbed and translated into chemical reactivity. Importantly, increasing light intensity does not necessarily improve performance: excessive photon flux can promote side reactions, catalyst deactivation, or product degradation. Effective photochemistry therefore requires deliberate matching of light-source emission to photocatalyst absorption and careful control of photon dose rather than indiscriminate intensification.



The complexity of photon management increases further in multiphasic systems containing gases or solids. Gas−liquid interfaces introduce refraction and reflection due to refractive index differences, leading to photon losses in regimes dominated by large bubbles, while finely dispersed bubbles can instead redirect light and enhance local absorption. Solid photocatalysts introduce additional challenges by scattering light anisotropically while simultaneously participating in the reaction. Scattering redistributes photons within—and sometimes out of—the reaction medium, complicating mechanistic interpretation and making mixing and hydrodynamics critical design parameters.

Scaling photochemical transformations from laboratory to production scale demands the parallel scaling of photon supply. Increasing optical power introduces challenges related to heat dissipation, nonuniform irradiation, and reactor design. Treating photons as reagents, quantified in equivalents relative to the substrate, provides a unifying framework for identifying photon-limited regimes and distinguishing them from limitations imposed by intrinsic kinetics or mass transfer. Systematic variation of wavelength and intensity not only enables robust scale-up but also yields mechanistic insight by revealing rate-limiting steps in multicomponent catalytic cycles.

In this Account, we describe how photon control, characterization, light interactions, and photoreactor engineering together define the efficiency, reproducibility, and scalability of photochemical processes. In addition, we discuss fundamental photonic principles for photochemistry and highlight strategies that enable predictable, selective, and industrially relevant photochemistry across reaction conditions and scales.

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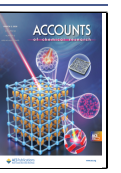

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INTRODUCTION

Light has long captivated scientists in academia and industry because of its unique ability to initiate and steer chemical transformations.[5−9] Today, photochemical technologies span a wide range of applications, including fine-chemical synthesis, wastewater treatment, carbon dioxide conversion, and water splitting.[10−13] The use of photons exemplifies sustainable processing, aligning perfectly with the principles of green chemistry,[14] while the mild reaction conditions provide access to previously inaccessible reactivities, which is particularly valuable in late-stage functionalization.[15,16] These advantages continue to drive the adoption of photochemical methods across diverse areas of chemistry.

At the heart of every photochemical transformation is the absorption of light, where a single photon excites a single molecule according to the Grotthuss−Draper and Stark−Einstein laws.[6] The probability of this excitation depends on both the energy of the incident photons and the electronic structure of the absorbing species. Some organic molecules undergo direct photochemistry,[17] with small aromatic molecules typically absorbing high-energy UV-C light, whereas larger conjugated structures, such as those found in many pharmaceutical intermediates, absorb at longer wavelengths (UV-A to visible).[18] Photocatalysts expand this reactivity landscape by decoupling absorption from substrate structure and enabling highly controlled transformations.[19−22] Upon excitation, photocatalysts can engage in oxidative or reductive single-electron transfer (SET), proton-coupled electron transfer (PCET), or energy-transfer pathways that generate reactive intermediates such as singlet oxygen.[23−26] Additional modes of reactivity such as hydrogen atom transfer (HAT) and halogen atom transfer (XAT) further extend the toolbox of radical chemistry.[27−29]

The merger of photocatalysis with transition metal catalysis has opened powerful new avenues for bond construction.[4,30−32] These dual catalytic cycles often require precise coordination of fast, photon-driven events with slower organometallic steps. For example, in nickel-catalyzed cross-couplings, photon-induced processes typically proceed much more rapidly than oxidative addition or reductive elimination.[33,34] If these rates are not aligned, the overall transformation may become limited by specific mechanistic steps, leading to catalyst deactivation or diminished efficiency.[35] Efficient mass transfer is similarly essential to ensure delivery of reactive species to catalytic sites.[36−38]

Despite the central role that photons play in enabling reactivity, the proper supply of light is often overlooked. The intensity, wavelength, and total number of photons delivered to a reaction directly determine whether a process operates in a photon-limited regime or suffers from overirradiation, both of which can compromise efficiency, selectivity, or catalyst stability.[17,39,40] A rigorous understanding of photon supply, i.e., how photons are transported, supplied, absorbed, and utilized, therefore becomes indispensable for both reaction optimization and scale-up.

In this Account, we highlight recent advances in understanding and engineering photon delivery in photochemical organic synthesis. We emphasize how reactor selection, characterization, and the systematic screening of irradiation conditions together shape reaction performance across laboratory and production scales.

PERFECT PITCH: TUNING THE PHOTON SUPPLY

For any photochemical reaction to proceed, photons must be absorbed; thus, achieving spectral overlap between the photocatalyst and the light source is essential. LEDs have become the preferred light sources in synthetic photochemistry due to their wavelength flexibility, narrow emission profiles, energy efficiency, ease of integration and relative low cost.[41,42] Despite their narrow peaks, LED emissions still span a distribution of photon energies, and photocatalysts may absorb outside the primary maximum of the source. Thorough characterization of both the emission spectrum and the photocatalyst absorption profile is therefore crucial for understanding and controlling reactivity.[43−45]

Tuning the wavelength of irradiation provides a powerful handle for adjusting photocatalyst redox states and steering reaction outcomes.[46−48] In some systems, changing the wavelength can modulate photon absorption without altering the fundamental pathway (Figure 1a). This was demonstrated by Pieber and co-workers in their study of dual catalytic C−N cross-couplings using carbon nitride and nickel.[35] As shown in Figure 1b, using green light instead of blue, where the photocatalyst has lower absorbance, reduces photon absorption and slows the photochemical steps to match oxidative addition. This alignment of rates suppressed catalyst deactivation, increased average yields, and significantly improved reproducibility. Wavelength choice also plays a critical role in controlling side reactions. In the synthesis of aryl ketones, irradiation at ∼390 nm promoted product formation, but then, under continued irradiation, also degradation of a subset of products in a Norrish type II cleavage.[49,50] Shifting the irradiation wavelength to ∼456 nm completely shut down this pathway, as the new carbonyl bond is no longer excited, while still permitting product formation.

Modulating photon flux is not limited to changing light sources. Dimmable LEDs allow direct adjustment of intensity with minimal changes to spectral distribution (Figure 1c). Although light dose is often described as intensity × time,[6] varying these parameters proportionally does not guarantee identical outcomes. Light intensity can influence product distributions, mechanistic regimes, and observed kinetics.[51,52] In semiconductor photocatalysis, for example, high-intensity irradiation increases charge-carrier recombination, lowering efficiency.[53]

The need to tailor photon flux to individual transformations was further highlighted by a self-optimizing robotic platform for photocatalytic oxytrifluoromethylations.[54] The Bayesian

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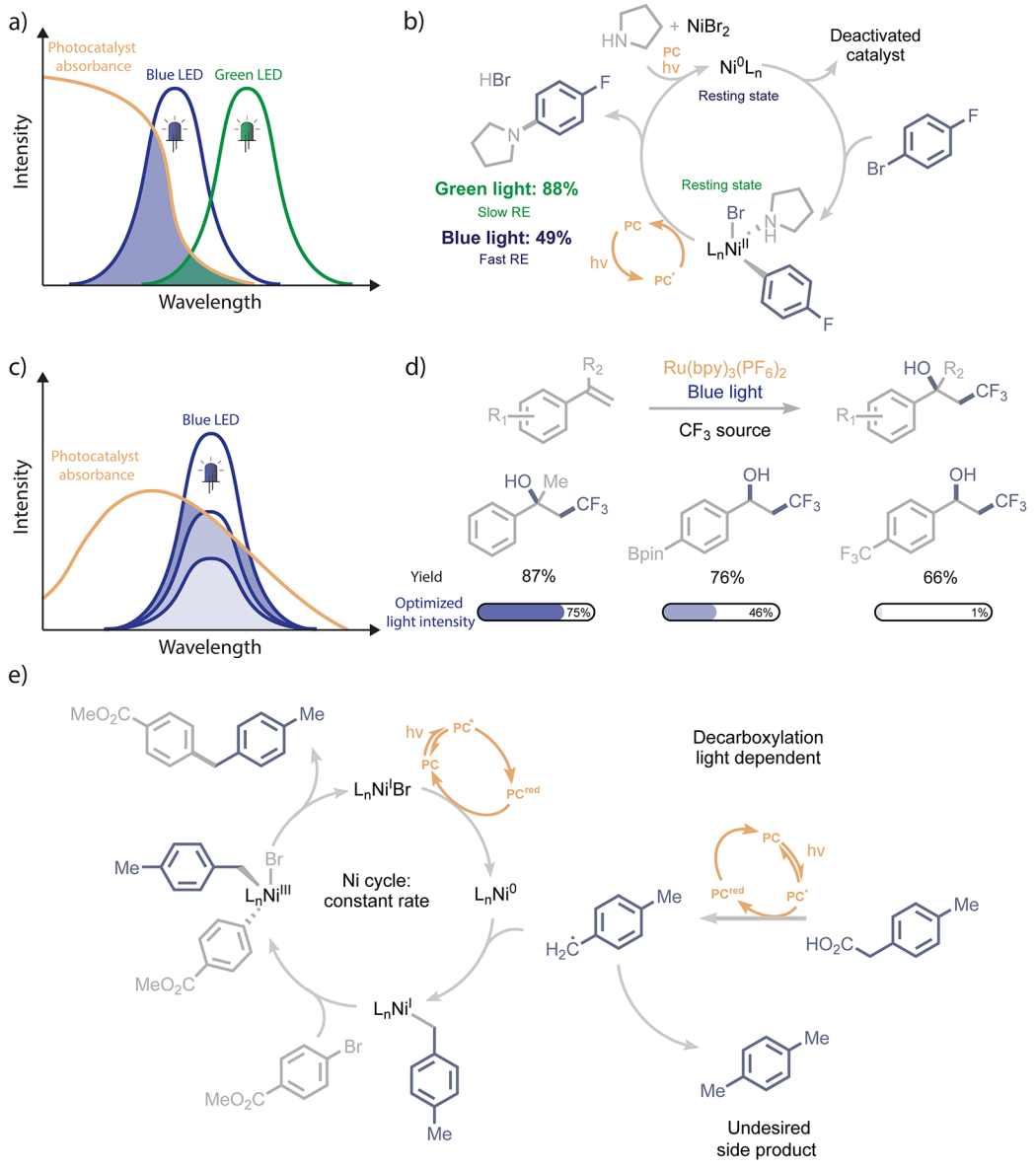

Figure 1. Tuning wavelength and photon flux to control photochemical reaction outcomes. (a) Schematic representation of the absorbance spectrum of a photocatalyst and the emission profiles of blue and green LEDs, illustrating how spectral overlap governs photon absorption. (b) Simplified mechanism of a dual catalytic C−N cross-coupling, showing how reduced photon absorption under green-light irradiation slows down reductive elimination (RE) (PC, photocatalyst; RE, reductive elimination), matching rates and preventing catalyst deactivation.[35] (c) Absorbance spectrum of a photocatalyst overlaid with the emission profiles of a dimmable blue LED at different intensities, demonstrating modulation of photon flux with minimal changes in spectral distribution. (d) Selected substrate scope of a photocatalytic oxytrifluoromethylation of alkenes, highlighting substrate-dependent optimal light intensities.[54] (e) Proposed mechanism of a decarboxylative C−C coupling, illustrating how photon absorption controls the rate of radical generation relative to the nickel catalytic cycle.[4]

optimization algorithm consistently identified optimal intensities far below the maximum optical output, often varying from 75% down to 1% depending on the substrate (Figure 1d). Excessive light intensity could initiate unproductive or degradative pathways, underscoring the substrate-specific nature of photon demand.

Matching the rate of radical generation to subsequent steps is equally important in multiphoton or dual catalytic processes. In a decarboxylative $C(sp^2)-C(sp^3)$ coupling enabled by graphitic carbon nitride (gCN), shorter-wavelength irradiation accelerated radical formation beyond the capacity of the nickel catalytic cycle, generating undesired photodecarboxylation products (Figure 1e).[4] A similar imbalance appeared during scale-up: using three lamps produced radicals faster than they could be consumed, whereas reducing intensity, effectively tuning photon supply, restored productive coupling.

Beyond screening photon flux and intensity, quantifying these parameters offers valuable insight into photon-limited regimes, underlying reaction mechanisms, and scale-up opportunities.[55,56] Expressing photon dosage in terms of photon equivalents becomes particularly useful in multiphoton processes,[57,58] where the stoichiometric requirement for absorbed photons is nonintuitive and can directly influence reaction kinetics and scale-up strategies. The quantum yield, defined as the ratio of product formed to the amount of photons absorbed, provides details on reaction efficiency and

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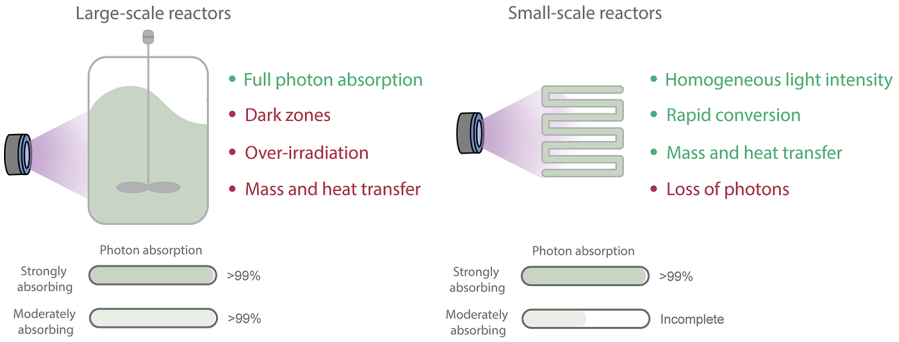

Figure 2. Comparison of photon absorption and irradiation characteristics in large-scale and small-scale photoreactors. Large reactors exhibit long optical path lengths that promote near-complete photon absorption but lead to dark zones and overirradiation, whereas small-scale reactors provide more homogeneous irradiation at the expense of potential photon losses in weakly absorbing systems.

can reveal whether a reaction proceeds via an efficient radical chain mechanism.[56] This metric depends on several factors, including wavelength and light intensity.[59−61] However, nonproductive absorption, reaction kinetics and other rate limiting steps can result in misleading quantum yield values, since excess photons may be absorbed without contributing to product formation.[62,63] Due to these effects, experimentally determined quantum yields are typically considered "effective" quantum yields, rather than intrinsic.[1] Although photons are inexpensive (<$1 per mole), their consumption becomes economically significant at scale, motivating interest in high quantum-yield processes, even when product yields are moderate.[64]

Chemical actinometry remains the most widely used method for determining incident and absorbed photon fluxes across wavelengths.[65,66] By performing a reference reaction with a known quantum yield, the number of absorbed photons can be inferred from product formation. Alternatively, spectroradiometry provides information by directly measuring incident light intensity.[67,68] To resolve spatial variations in light intensity and photon absorption within a reactor, more sophisticated tools such as ray-tracing simulations or solutions to the radiative transport equation (RTE) are required.[69−72] These approaches rely heavily on accurate input data, such as light-source emission characteristics, refractive indices, and material optical properties, and must be experimentally validated before being used.[1,73]

PHOTOREACTOR SYSTEMS: HOLD ON TO YOUR LIGHT

The number of photons reaching a reaction mixture is determined not only by the light source but also by the photoreactor and any intervening materials, such as reflectors, that influence photon transport.[1,74] These components can be collectively referred to as the photoreactor system, and any variation within the system can alter the number of incident photons, requiring recharacterization. Positioning the light source relative to the reactor and using suitable reflectors can enhance the light intensity reaching the reaction mixture. The fraction of incident photons absorbed in the system depends on the reaction medium, the concentration of absorbing species, and the distance the light travels through the mixture, as described by the Lambert−Beer law.[5]

A wide range of photoreactor designs has been introduced for synthetic photochemistry, yet the absence of standardized equipment remains a major barrier to reproducibility.[8,75] Variations in local light intensity, wavelength, and heat management across photoreactor systems emphasize the importance of characterizing the light source and photon parameters within the reactor.[76] For example, repositioning the light source relative to the reactor was shown to dramatically impact space-time yields.[77] In the photo-Favorskii rearrangement, different reactor−light source configurations produced markedly different outcomes. This effect arose from changes in the optical path length as well as the evolving absorbance properties of the reaction medium: the starting material exhibits stronger light absorption than the product, causing the overall absorption characteristics to shift over the course of the reaction.

In large batch reactors (>1 L), typical of early industrial scale-up, the long optical path length drives the transmittance toward zero, such that nearly all incident photons are absorbed, even for only moderately absorbing media (Figure 2). Complete absorption of photons inevitably creates pronounced dark zones within the reactor volume. As a result, reaction kinetics become highly heterogeneous: near the reactor walls, the high local light intensity leads to rapid photochemical turnover, whereas at the center, few photons penetrate and the kinetics are essentially zero. This imbalance causes prolonged exposure of product molecules at the illuminated periphery to irradiation until reaction completion. Overirradiation can cause degradation, side reactions, and diminished selectivity. Compounding these issues, large reactors can also suffer from poor mass and heat transfer and limited process control, further complicating scale-up.

Miniaturization can be used to achieve a homogeneous light intensity throughout a reactor. As a result, continuous-flow photoreactors are now frequently employed in photochemical processes. The small dimensions of these reactors make photon absorption sensitive to the absorption properties of the reaction medium, potentially leading to photon transmittance and losses if photons are not reflected back into the reactor volume (Figure 2). When properly engineered, homogeneous irradiation eliminates dark zones and enables continuous product formation while minimizing overirradiation. Their high surface to volume ratios also yield superior heat dissipation.[78,79] Operating such reactors in recirculation mode serves as a complementary technique that leverages these properties, though repeated exposure of product to the irradiation zone may still promote overirradiation and secondary reactivity.

Characterizing photon transport in small-scale reactors presents its own challenges. While chemical actinometry can quantify photon flux, its results depend strongly on the

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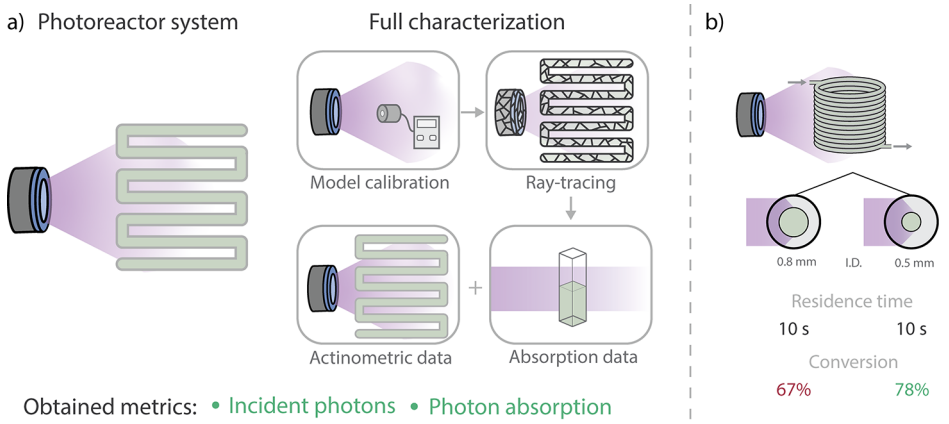

Figure 3. Workflow for the full photonic characterization of a photoreactor system (a) and its impact on photon absorption (b). Ray-tracing, actinometry, and absorption measurements are combined to quantify incident and absorbed photons, revealing light-concentration effects and effective optical path lengths in capillary reactors of different inner diameters.[2,74]

absorption characteristics of the reaction mixture, making measurements nontransferable across conditions.[80] The Lambert–Beer law provides a more generalizable description by relating absorbance to extinction coefficient, concentration, and optical path length,[81] but introduces the optical path length as an additional unknown that must be determined. As a result, accurate quantification typically demands a combination of characterization techniques.[1,82]

It is important to realize that determining optical path length is complicated by reflection, refraction, and the nonuniform emission profiles of real light sources. Even small uncertainties can substantially affect the calculated fraction of absorbed photons. To address this, a workflow integrating validated modeling tools with targeted experiments was developed to characterize three reactor geometries.[1] Digital twins of each photoreactor were constructed, and ray-tracing simulations were used to determine incident photon flux. These values were then paired with kinetic data from potassium ferrioxalate actinometry[80,83] to extract an *effective* optical path length enabling clear distinction between incident and absorbed photons (Figure 3a).

Interestingly, the experimentally determined path lengths were larger than commonly assumed, underscoring the importance of thorough characterization. Reflection and refraction effects can cause multiple passes through the reaction mixture, thereby increasing the effective optical path length. Moreover, ray-tracing simulations of capillary reactors revealed that refraction by the capillary causes light to concentrate on the reactor volume.[2,84] Reflection and refraction occur at interfaces where there is a change in refractive index ($n_{air} \approx 1$ and $n_{capillary} \approx 1.3$) and are described by the Fresnel equations.[85] This concentration effect was used to increase the space-time yield of photoreactor systems, as the number of incident photons per reactor volume can be significantly increased by using capillaries with narrower inner diameters (Figure 3b).

FULL THROTTLE: ACCELERATION BY ADDING GAS

The use of gaseous reagents in organic synthesis offers several advantages, including improved atom economy, access to new reaction pathways, and simplified downstream processing.[86,87] In batch reactors, gases are typically introduced by bubbling or as headspace, resulting in limited gas–liquid contact. This becomes particularly problematic when the gas acts as a reagent that must rapidly quench photocatalytically generated radical intermediates in the liquid phase. If gas–liquid mass transfer cannot keep pace with radical reactivity, unproductive or degradative side products are often formed. Increasing the interfacial area between phases enhances gas–liquid mass transfer, a key motivation for the development of gas–liquid continuous-flow reactors. These systems not only provide improved process control and inherently safer operation but also give rise to distinct gas–liquid flow regimes depending on flow rates and reactor geometry.[88,89] For example, bubbly flow is characterized by small, spherical bubbles, whereas slug flow involves gas bubbles that span nearly the full axial length of the reactor.

Gases do not need to be reactive to be beneficial. Introducing an inert gas can enhance mixing within the liquid phase and improve overall reactor performance. Slug flow, for instance, forms characteristic toroidal vortices within each liquid segment, which can maintain solids in suspension. This strategy enabled selective fluorination of phenoxyacetic acid derivatives using a modified carbon nitride catalyst.[90] Enhanced mass transfer arising from these vortices can also be exploited in hydrogenations or oxidations using gaseous reagents.[91–93]

Multiple gases can be used synergistically to drive efficient photochemical transformations. A notable example is the C–H functionalization of light alkanes using carbon monoxide under UV irradiation (Figure 4a).[94] Although gaseous hydrocarbons are inexpensive and abundant, their high C–H bond dissociation energies pose a challenge. With a suitable photocatalyst and ~365 nm light, however, such transformations become feasible. In this system, the photocatalyst, a Michael acceptor, and dissolved gases are present in the liquid phase, while the dissolved gas concentration follows Henry's law. A continuous-flow setup allows safe pressurization up to 52 bar, enabling higher dissolved gas concentrations and, in some cases, liquefaction of gaseous reagents. Under these conditions, even methane, ethane, and butane can be efficiently functionalized.

Beyond mass transfer effects, introducing a gas phase can influence photon transport and thereby affect photochemical performance.[95–97] Gas–liquid interfaces induce scattering,

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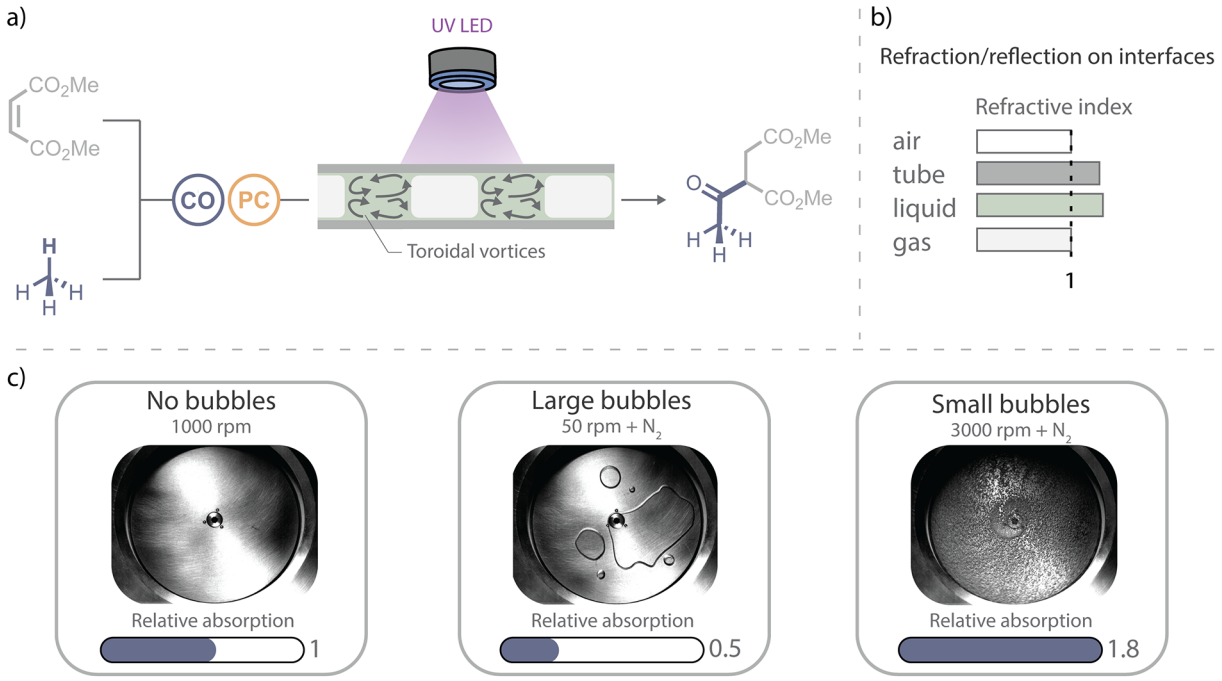

Figure 4. Influence of gas–liquid interfaces on photon absorption in photochemical reactors. (a) Continuous-flow photocatalytic carbonylation of methane in a slug-flow regime[94] and (b) the differing refractive indices of the materials. (c) Representative images from a pRS-SDR illustrating how bubble size and hydrodynamics alter relative photon absorption.[2]

refraction, and reflection due to the refractive index difference between gas and liquid (Figure 4b).[69,98,99] Most gases have refractive indices close to 1 and minimal absorption in the UV-A and visible regions. Computational studies of uniformly dispersed spherical bubbles show that light encountering small spherical bubbles is primarily scattered forward, typically exiting the bubble on the opposite side.[100−102] At low gas fractions and small bubble sizes, these scattering events have only a modest impact on overall photon absorption.

Photon absorption becomes more complex in nonuniform or dynamically evolving gas–liquid regimes. Full photonic characterization, using the workflow described in Figure 3a, was applied to two continuous-flow reactors to examine these effects.[2] In slug flow, photon losses in the liquid phase were relatively small. However, studies in a photo rotor–stator spinning disk reactor (pRS-SDR) revealed a richer interplay between bubble size, photon transport, and reaction performance. Large bubbles were found to reduce photon absorption within the liquid. Notably, as illustrated in Figure 4c, smaller bubbles increase photon absorption, likely due to additional scattering and modified light trajectories created at gas–liquid interfaces. Under high shear conditions, these effects can concentrate light within the liquid phase, potentially accelerating photochemical reactions.

LIGHT SCATTERING: MAINTAINING SOLID PERFORMANCE

Solids are widely used in chemical processes as heterogeneous additives, often dispersed within the reaction medium. Interest in heterogeneous photocatalysts has grown rapidly in the synthetic community after decades of use in photocatalytic water splitting and environmental remediation.[103−106] The benefits of these cheap and sustainable photocatalysts in comparison to typical homogeneous options lie in their easy removal from product streams, their minimal CO_2 footprint, and their recyclability.[4,107−109] TiO_2 slurries in particular have been well studied for applications ranging from wastewater treatment to synthetic oxidations.[110−116] Other metal oxides have also been used in synthesis, where they typically promote oxidation processes.[117,118] Recently, graphitic carbon nitride materials (gCNs) have emerged as broadly applicable heterogeneous photocatalysts, enabling oxidations, C–heteroatom couplings, and various C–C bond-forming processes.[4,50,107,109,119−126]

Semiconductors generally operate as photocatalysts that can engage in multiple pathways after absorbing light. Photons with sufficient energy promote electrons from the valence band to the conduction band, generating electron–hole pairs (Figure 5a).[13] These charge carriers can migrate to the surface to engage in redox or energy transfer reactions or unproductively recombine in the bulk of the material.[127,128] The rate of generation and recombination of electron–hole pairs strongly influences the reaction kinetics, and recombination should be minimized for optimal reaction efficiencies.[129]

Apart from absorption, the addition of solids adds extra complexity as light scatters within the medium (Figure 5a). Scattering redistributes the incoming light into all directions of space, albeit commonly at varying intensities (anisotropic).[129] It is governed by the interfacial properties between the solid and the surrounding liquid, which in turn depend on factors such as particle composition, size distribution, surface area, and porosity.[130] The properties of solids in dispersion are altered by reaction conditions, as variations in pH, temperature, or stirring can all lead to differing interaction of the catalyst with incident light.[131,132] These effects are not confined to (semiconductor) photocatalysts and should likewise be taken into account for other solid materials.

Studies have shown that scattering dominates over absorption for a range of TiO_2 photocatalysts and wavelengths.[131,133] Scattering can result in photons leaving the

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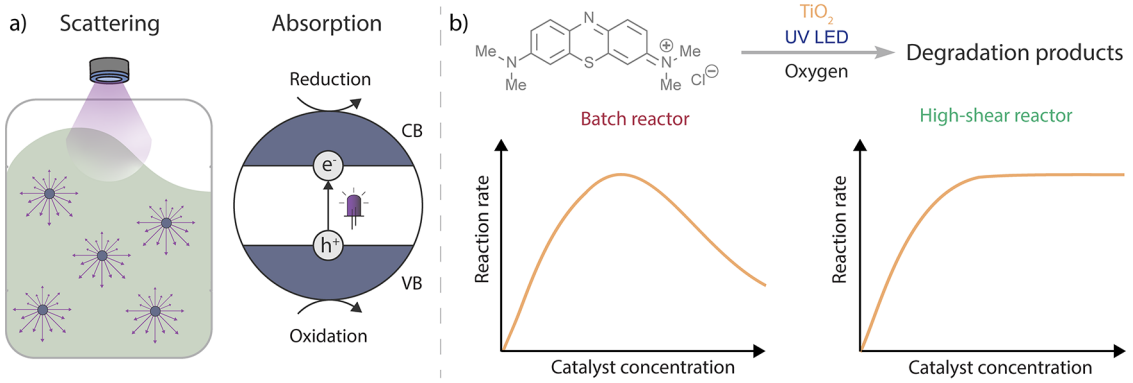

Figure 5. Effect of light scattering and mass transfer on heterogeneous photocatalysis. (a) Dispersed solids induce anisotropic light scattering while semiconductor photocatalysts absorb photons to generate charge carriers (VB, valence band; CB, conduction band). (b) Excessive catalyst loading reduces performance in batch reactors but is mitigated under high-shear conditions due to improved mixing and photon utilization.[108]

medium or being absorbed by competing species, leading to less efficient utilization of light.[134,135] Nonetheless, these losses can be mitigated if the scattered photons are subsequently absorbed by the photocatalyst and engage in the reaction. Neglecting scattering effects and assuming identical absorbed light intensities renders direct rate comparisons largely meaningless, as even minor changes may considerably alter the system.[136] A rigorous analysis must account for the optical properties of the system before drawing conclusions about intrinsic photocatalytic activity.[133]

To correct for the loss of absorbed light via scattering, studies have shown both an increase in reactor depth and an increase in photocatalyst concentration to be effective.[137] However, these adaptions have to be matched to the use case: If there are competitively absorbing species present the benefit of reactor depth vanishes. Scattering does not always imply losses of photons as the use of a scattering medium within a reactor can be used to elongate photon path lengths and so increase photon absorption.[138]

The interplay between photocatalyst concentration, mass transfer, and photon absorption was demonstrated in the degradation of methylene blue using TiO_2 in both a batch reactor and a pRS-SDR.[108] The pRS-SDR employs a rapidly rotating disk positioned between two stators, generating high shear forces due to velocity gradients.[139,140] Such hydrodynamics make it well suited for processes requiring intense mass transfer. In a batch reactor, reaction rates increased linearly with light power at low intensities, consistent with a photon-limited regime.[5] At higher intensities, the dependence became nonlinear as mass transfer and photocatalyst concentration began to limit the reaction. A maximum rate was observed at an intermediate TiO_2 loading, with higher concentrations causing decreased rates due to strong attenuation in the opaque slurry and poor mixing. In contrast, the pRS-SDR exhibited dramatically higher reaction rates at equivalent light power to reactor volume ratios. Its superior mixing alleviated the limitations observed in batch by rapidly transporting photocatalyst particles back into illuminated regions, improving gas–liquid mass transfer, and ensuring uniform catalyst dispersion (Figure 5b).

Similar effects were also discussed by the team of AbbVie in their scale up of a homogeneous iridium-catalyzed N-arylation.[141] They observed an initial increase of the reaction rate, followed by a gradual decline as they further increase the photocatalyst loading. They argue that at high photocatalyst loadings all the light is absorbed in a minimal fraction of the volume whereas the quenching species is distributed throughout the entire reactor volume, creating a lower effective ratio of intended reaction partner to excited state photocatalyst. These observations highlight the need to tailor photon absorption to the reaction system by considering both the absorption characteristics of the medium and the optical path length.

In heterogeneous systems, solutions to the RTE must consider the scattering coefficient and the phase function of the scattering coefficient in addition to the absorption coefficient, typically requiring assumptions that simplify the problem.[142] This commonly includes homogenization of the mediums properties (particle size, absorption coefficient) or reduction of the anisotropy of scattering to a two- or six-flux system (proposing that all scattered photons travel forward and backward or in the directions of the Cartesian coordinates, respectively).[113,129,143,144] Alternative techniques like ray tracing also require assumptions on the shape and distribution of the solids, which might cause discrepancies with real-life situations.[1,145]

PUMP UP THE VOLUME: INCREASING PHOTON FLUX AND PRODUCTIVITY

Scaling photochemical processes places demands on reactor design and photon delivery that are distinct from those encountered in thermal chemistry.[6,43] Conventional scale-up strategies rely either on increasing reactor dimensions or on numbering-up identical units.[5] In photochemistry, continuous-flow reactors, particularly micro- and milliflow systems, have become the preferred approach, as they have a small footprint while enabling continuous production. The same principles apply to light sources: photon supply can be increased either by numbering-up multiple (identical) lamps or by deploying higher-power light sources to deliver greater photon flux to a single reactor (Figure 6a).[3,74] In purely photon-limited systems, scaling productivity may therefore require little more than increasing the number of incident photons while keeping reactor geometry unchanged. However, excessive intensification risks overirradiation, side product formation and may introduce new practical limitations.

Increasing photon flux inevitably raises challenges related to heat management. Reaction mixtures can warm due to nonradiative relaxation following photon absorption in the reaction medium or through interactions between light and

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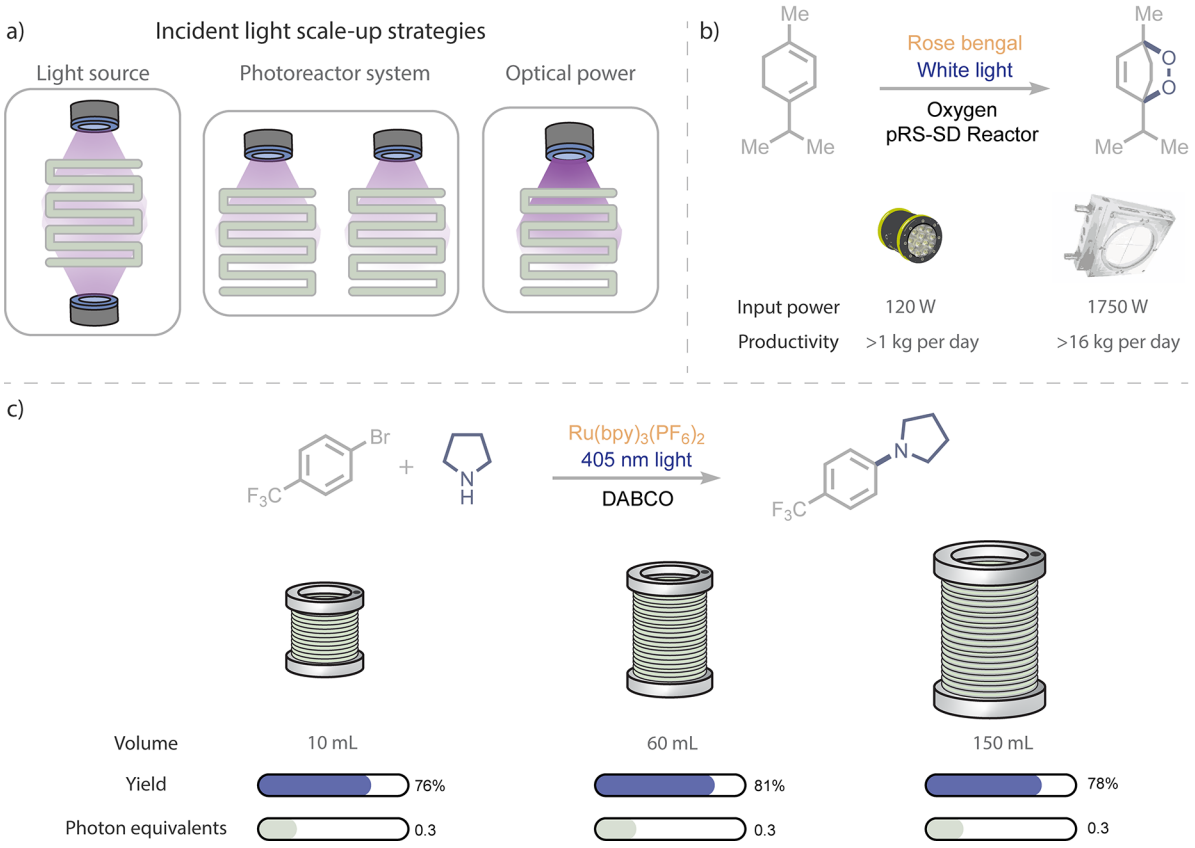

Figure 6. Strategies for increasing photon flux and productivity in photochemical scale-up. Incident light can be intensified by (a) numbering-up light sources or reactors or (b) increasing optical power.[3,140] (c) Scale-up of a metallaphotoredox C−N coupling using absorbed photon equivalents as the scaling parameter, enabling consistent yields across reactor volumes.[55]

reactor materials. In the context of scale-up, the number of photons absorbed, rather than merely incident, becomes a critical design parameter. Treating photons analogously to chemical reagents by expressing photon equivalents relative to substrate loading provides a useful framework for scale-up.[146] Using this approach, consistent yields were achieved for a photocatalytic C−N cross-coupling reaction across multiple reactor systems with different scales and operating modes by tuning the reaction/residence times to provide comparable photon equivalents (i.e., photon equivalents ∝ absorbed photon flux × time). This highlights the importance of photonic characterization in both continuous-flow and batch processes (Figure 6c).[55] Nevertheless, this strategy is most effective under photon-limited conditions and may yield divergent outcomes when other kinetic or mass-transfer limitations dominate.

Scaling photochemical reactions that involve solid additives (catalyst, base, or other solid reagents or products) introduces additional constraints. Slurries require sufficient agitation to prevent sedimentation and ensure uniform irradiation, yet narrow reactor dimensions are often favored for their excellent heat transfer and high surface to volume ratios. As discussed earlier, the pRS-SDR offers a viable solution for scaling heterogeneous photochemical systems.[108] Its intense mixing regime mitigates limitations that commonly restrict productivity in conventional reactors.[147]

This capability was demonstrated during the scale-up of the photochemical oxidation of α-terpinene to ascaridole.[140] Operation of the pRS-SDR under photon-limited conditions enabled production rates of approximately 1 kg/day. Recognizing the strong dependence of productivity on photon flux, the reactor was subsequently equipped with a more powerful light source to increase photon delivery.[3] This intensification resulted in higher operating temperatures, though short-term studies indicated only minor negative effects, likely arising from reduced oxygen solubility rather than thermal degradation.

Systematic variation of light-source power and rotor speed revealed distinct photon-limited and mass-transfer-limited regimes.[147] At low rotational speeds, increasing photon flux shifted the system toward mass-transfer limitation, whereas at higher rotational speeds both parameters influenced productivity. Under optimized conditions, the reaction approached intrinsic kinetic control and achieved productivities exceeding 16 kg/day (117 mol/day) (Figure 6b). In contrast, when the same reactor was applied to the photooxidation of β-citronellol, a reaction frequently used to benchmark photoreactors,[148,149] productivity plateaued beyond 1.27 W/cm^2 and 2000 rpm. This insensitivity to further intensification indicates kinetic limitations, such as a high-energy transition state, rendering the reaction unsuitable for benchmarking under these conditions.

Several alternative reactor concepts have been developed to address scale-up challenges in photochemistry by balancing photon supply, surface to volume ratio, and process continuity. Leblebici and co-workers introduced a monolithic reactor design that maximizes light utilization and enables efficient gas−liquid photochemistry at scale, achieving energy efficiencies up to 2 orders of magnitude higher than conventional systems.[150] George and co-workers reported a Taylor vortex

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flow reactor for the scale-up of a metallaphotoredox C−O coupling.[151] The narrow annular flow path and central rotor generate Taylor vortices that provide excellent mixing; after suppressing precipitate formation, this system delivered productivities extrapolating to multiple kilograms per day.

A fundamentally different strategy was demonstrated by Wu and co-workers using a high-speed recirculation setup.[152] Recirculation decouples reaction time from reactor volume and flow rate, allowing high flow rates without sacrificing reaction time. Careful optimization of tubing diameter and flow conditions produced a stable, uniformly irradiated suspension with no clogging from solid sedimentation. Although recirculation does not eliminate overirradiation, improved heat and mass transfer resulted in higher selectivity and productivity. This automated system significantly outperformed batch photoreactors and enabled transformations on the 100 g to 1 kg scale using mesoporous graphitic carbon nitride as the photocatalyst.

CONCLUSION AND OUTLOOK

The control and quantification of photons in photoreactor systems are essential for achieving efficient, reproducible, and selective photochemical transformations. A growing body of work demonstrates how light intensity, wavelength, and photon flux influence reaction outcomes, providing valuable insight into reaction kinetics, limiting steps, and photon utilization. Crucially, photon absorption cannot be understood solely from the properties of the photocatalyst or reaction medium; it is equally governed by the characteristics of the photoreactor system and the light source, which together define how photons are delivered, redistributed, and ultimately absorbed.

Substantial progress has been made in the development of energy-efficient light sources covering a broad spectral range and offering scalable output powers. These advances enable photochemical investigations across scales spanning milligrams to kilograms.[3] Commercial light sources are increasingly accompanied by detailed characterization data, supporting improved reproducibility and standardization. In contrast, custom or nonstandard light sources still require careful experimental characterization to determine optical power, spectral distribution, and effective intensity within the reactor.[68,76,153] Continued efforts toward standardized reporting of photon-related parameters will be critical for meaningful comparison between studies and for accelerating translation to larger scales.

Multiphasic photochemical systems involving gases and solids present both opportunities and challenges. Gas−liquid and solid−liquid interfaces introduce scattering, refraction, and reflection phenomena that alter photon pathways in nontrivial ways. While these effects can lead to photon losses, they can also be exploited to enhance absorption through careful control of operating conditions, flow regimes, and mixing.[2] In heterogeneous systems, photocatalyst concentration, dispersion quality, and hydrodynamics strongly influence the reaction outcome, underscoring the need to consider optical and transport phenomena alongside chemical reactivity when designing photochemical processes.

The systematic characterization of light−matter interactions has proven its importance in modern photochemistry in organic synthesis, particularly for scale-up.[154] Photoreactor setups can be engineered to maximize photon efficiency and promote homogeneous irradiation, as exemplified by continuous-flow systems.[64,155] Given the complexity of photon transport in realistic reactor geometries, computational tools such as ray-tracing and radiative transport modeling are becoming indispensable for evaluating photoreactor performance, guiding reactor design, and enabling predictive scale-up.

Looking ahead, the integration of rigorous photon management with automation, digital twins, and machine learning-driven optimization is poised to further transform photochemical process development.[156] As photons increasingly come to be treated as quantifiable reagents rather than abstract energy inputs, photochemistry will continue to mature into a robust, scalable, and industrially relevant platform for sustainable synthesis.

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Notes

The authors declare no competing financial interest.

Biographies

Jasper H. A. Schuurmans (born 1998, Oss) obtained his BSc and MSc degrees in Chemical Engineering from Eindhoven University of Technology. He subsequently joined the Noël Research Group at the University of Amsterdam as a PhD candidate, where his research focuses on photoreactor modeling, scale-up, and process intensification.

Florian Lukas was born in 1993 in Vienna. He received his MSc in Technical Chemistry from the Technical University of Vienna in 2022 after working on the development Claisen-type rearrangements and exploring $C(sp^3)$-$C(sp^3)$ bond-formation via hydrazones and photo-HAT in the Noël group. After graduating with distinction, he joined Prof. Noël's group as a PhD candidate working on use of heterogeneous photocatalysts for decarboxylative chemistry and the development of platforms for automated experimentation.

Prakash Chandra Tiwari completed his master's degree in 2021 at IISER Bhopal, where he carried out research on Pd/NBE relay catalysis under the guidance of Prof. Manmohan Kapur. Following his graduation, he joined Prof. Debabrata Maiti's group at the Indian Institute of Technology Bombay (IIT-B) as a project research assistant. During his time in the DM lab, his work focused on palladium-catalyzed enantioselective C−H functionalization of free

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acids. He is currently a third-year graduate student at the University of Amsterdam in Prof. Noël's group, where his research centers on photocatalytic (sp^3)−H activation using gaseous reagents to enable rapid access to new chemical space.

Timothy Noël is a Full Professor and Chair of Flow Chemistry at the University of Amsterdam, where he focuses on the delicate synergy between synthetic organic chemistry and technology. He has received several awards for his research in flow chemistry, including the DECHEMA prize (2017), the Hoogewerff Jongerenprijs (2019), the IUPAC-ThalesNano prize (2020), the KNCV Gold Medal (2021), the ACS Sustainable Chemistry & Engineering Lectureship Award (2022), and the ChemSocRev Pioneering Investigator Lectureship (2023). In addition, he serves as president of the Flow Chemistry Society and is co-organizer of #RSCPoster.

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