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A reproducible integrating sphere method for standardised diffuse light characterisation of photoelectrochemical and photovoltaic devices

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Keywords

Solar water splitting, solar photovoltaics, diffuse irradiance, solar hydrogen production, solar fuels, photoelectrochemical systems

A reproducible integrating sphere method for standardised diffuse light characterisation of photoelectrochemical and photovoltaic devices

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Abstract

Testing photoelectrochemical and photovoltaic devices under realistic operating conditions is a critical phase of system development. However, most studies still focus on standard AM 1.5G tests at one sun (1000 W m^{-2}) irradiance with direct collimated light beams, which does not capture outdoor operation under broad angular illumination from diffuse skies and low solar elevation. Outdoor field trials are difficult to benchmark reproducibly due to the unpredictable nature of outdoor environments. Herein, we demonstrate a simple, laboratory-based method for generating reproducible, angularly broad “quasi-diffuse” illumination by coupling a continuous solar simulator to an integrating sphere. By positioning a photoelectrochemical device at the integrating sphere exit aperture, we produce a controlled quasi-diffuse illumination geometry and demonstrate its use in two case studies involving WO_3|$BiVO_4$|NiFeOOH photoanodes: one with a planar architecture and another coupled with a crossed-compound parabolic concentrator (CCPC) with wide-angle acceptance, designed to harvest diffuse and off-normal light. After normalisation to one-sun equivalent irradiance, the non-concentrating (planar) configuration performed similarly under direct and quasi-diffuse irradiance, whereas the CCPC-coupled configuration retained ≈ 30% of the direct-illumination photocurrent under quasi-diffuse conditions, consistent with acceptance-angle limited concentration. This method can be extended to any photoelectrode, photovoltaic or other photo-absorber, provided that its dimensions are compatible with the exit aperture of the integrating sphere. This work provides a practical, controllable and transferable framework for quasi-diffuse light characterisation that can be easily benchmarked across different research teams and will complement existing AM 1.5G measurements. This also offers a simple way to assess diffuse light tolerance, wide-angle acceptance and to validate ray tracing models using readily available laboratory equipment. This enables a straightforward test to assess the viability of device architectures in the laboratory before embarking on more challenging field trials.

Introduction

Competition in global renewable energy markets is rapidly driving down cost of solar technologies, with the economics of solar fuel production – either by electrolysis driven by photovoltaics (PV) or by photo-electrolysis using photo-electrosynthetic cells – becoming increasingly viable, particularly for decentralised or off-grid settings [1–5]. With the rapid expansion of technology, the importance of reliable, reproducible methods for characterising photo-absorbing materials such as photovoltaic cells or photoelectrodes is critical to enable continual development and improvement in photoelectrochemical (PEC) technology.

For photoelectrochemical systems, which will ultimately operate outdoors, the challenge is to replicate inherently variable weather conditions that can diverge greatly from controlled laboratory environments. Laboratory PV and PEC cells are typically characterised under standard “one sun” conditions with solar simulators that produce an irradiance of ≈ 1000 W m^{-2}. The spectral output is typically calibrated, or optical filters are used, to enable a spectral match close to reference Air Mass 1.5 Spectra, as defined by the American Society for Testing and Materials (ASTM) [6]. Although AM 1.5 is a very useful standard for comparing solar driven chemical and electrochemical reactions, it is applicable only to a single condition, which is a representative average of the U.S. states over the period of the year. Global irradiance levels, the proportions of direct to diffuse irradiance and cloud cover vary greatly over the course of the year. In many northern hemisphere climates, AM 1.5 is not representative of the solar resource for most of the year, as these regions frequently experience cloud cover that results in Lambertian scattering of the sun’s rays, so that they subsequently reach the surface of the photo-absorber over a wide range of incident angles. This is a challenging and complex phenomenon to replicate reproducibly in a laboratory.

Whilst the effect of different, single incident angles can be examined under a solar simulator, by using tilt tables or similar, solar simulators typically produce uniformly direct irradiance,

which therefore cannot reliably represent the effects of clouds that cause Lambertian scattering. With PV and PEC devices designed to operate at one sun, AM 1.5 conditions, shifts in the spectral output under partly cloudy or low solar elevation can drive the system away from their design operating points, which can have detrimental effects on both performance and stability [7,8]. Angular effects are already well recognised in the concentrating photovoltaics and concentrated PEC communities [9,10]. High concentration imaging optics systems such as parabolic reflectors can approach concentration ratios of several hundred to several thousand suns, but can typically only tolerate very limited incidence angle divergence from the normal in the order of 0.1 to 0.6°, meaning that very precise dual-axis tracking is required to maintain optical efficiency, and diffuse rays are largely rejected by the optical system [10–12]. Whilst low concentration, non-imaging optics can tolerate larger acceptance angles, beyond ray tracing simulations and experimental characterisation under direct irradiance at individual incident angles, there is limited analysis of the effects of angular or diffuse light [10].

Current methods to vary simulated sun intensity using solar simulators, involving beam attenuation via neutral density filters or lamp current adjustment, preserve a narrow, typically highly direct, angular distribution that cannot reproduce broad quasi-Lambertian distributions associated with cloudy or highly diffuse weather conditions. As a result, the response of photo-absorbers, electrochemical interfaces, PV and PEC systems remains poorly quantified in controlled laboratory settings. Illumination conditions in outdoor settings, whilst providing useful and real benchmarks, differ wildly between studies, with most focussing on clear sky periods that maximise device performance.

Herein, we address this methodological gap by introducing a simple, reliable and reproducible approach for simulating quasi-diffuse, angularly broad illumination in the laboratory. We achieve this by coupling a solar simulator to an angularly redistributing optical element that converts the collimated input beam into a broad angular distribution at the device plane. In this

work, we use an integrating sphere, typically employed as a spectrophotometry accessory, to perform this role. We use the term “quasi-diffuse” to describe illumination with a broad angular distribution approximating a Lambertian field, as produced by the multiple internal scattering events within the integrating sphere. This technique can be coupled with any photo-absorber, including single or multi-junction PV devices, PEC devices or PV-coupled PEC systems, provided the dimensions of the photo-absorber are compatible with the exit aperture of the integrating sphere.

For the solar fuels and PEC communities, this work provides a framework to emulate cloudy day operation in a controlled, repeatable laboratory test, without relying on weather dependent outdoor trials or access to dedicated field-testing facilities. For PV and materials scientists, this method offers a simple extension to standard solar simulator setups, using hardware that many laboratories already possess or can readily obtain, to quantify diffuse light tolerance of different devices and architectures. For those engineering solar optics, including non-imaging concentrators and light trapping structures, it enables the validation of wide-angle performance and ray tracing simulations under quasi-diffuse irradiation, using a reproducible protocol that can be replicated and compared across different laboratories.

In this study, we demonstrate the application of the method to a PEC reactor, with and without a non-imaging crossed-compound parabolic concentrator (CCPC). This work provides a framework to compare photo-absorber performance under direct and diffuse light conditions, complementing existing standards for one sun testing and supporting the development of realistic and comparable performance metrics for photo-absorber technologies operating in environments with higher proportions of diffuse irradiance.

Methodology

A WACOM Class AAA continuous solar simulator (model: WXS-210S-20) equipped with an AM 1.5 G filter was used to produce simulated sunlight in a controlled laboratory with ambient

temperature of 20 °C. Prior to any measurements, the Xe lamp was switched on and allowed to warm up and equilibrate for 30 minutes. A reference monocrystalline solar cell, generating a short circuit photocurrent of 127 mA at 1000 W m^{-2} irradiance, was used to calibrate the light intensity prior to measurements. The light spectrum of the solar simulator at 1000 W m^{-2} irradiance was measured using a StellarNet Black Comet UV/Vis (280 to 900 nm) and a StellarNet Dwarf Star NIR (900 to 1700 nm) spectrophotometer. A representative spectrum is shown in **Figure S1**.

Diffuse-like illumination was generated using an integrating sphere (UV-vis-NIR accessory, Bentham 150 mm diameter integrating sphere), with a Ba_2SO_4 diffuse coating providing high hemispherical reflectance across the UV-vis-NIR spectral range. The integrating sphere was equipped with a front entrance port that was aligned to the output beam of the solar simulator, as shown in **Figure 1a**. The photo-absorber receiving the diffuse irradiance was mounted at the circular exit aperture which had a diameter of ≈ 2 cm^2 (as shown in **Figure 1b**). **Figure 1c** provides an illustration of the first reflection of a single ray inside the integrating sphere. **Figure 1d-e** show a schematic drawing and photograph of the setup during the demonstration with the PEC reactor. This simple arrangement can be implemented using any light source with a known irradiance at the entrance port of the integrating sphere. Additional photographs of the setup with the PEC reactor mounted at the exit aperture of the integrating sphere are shown in **Figure S2** and **Figure S3**.

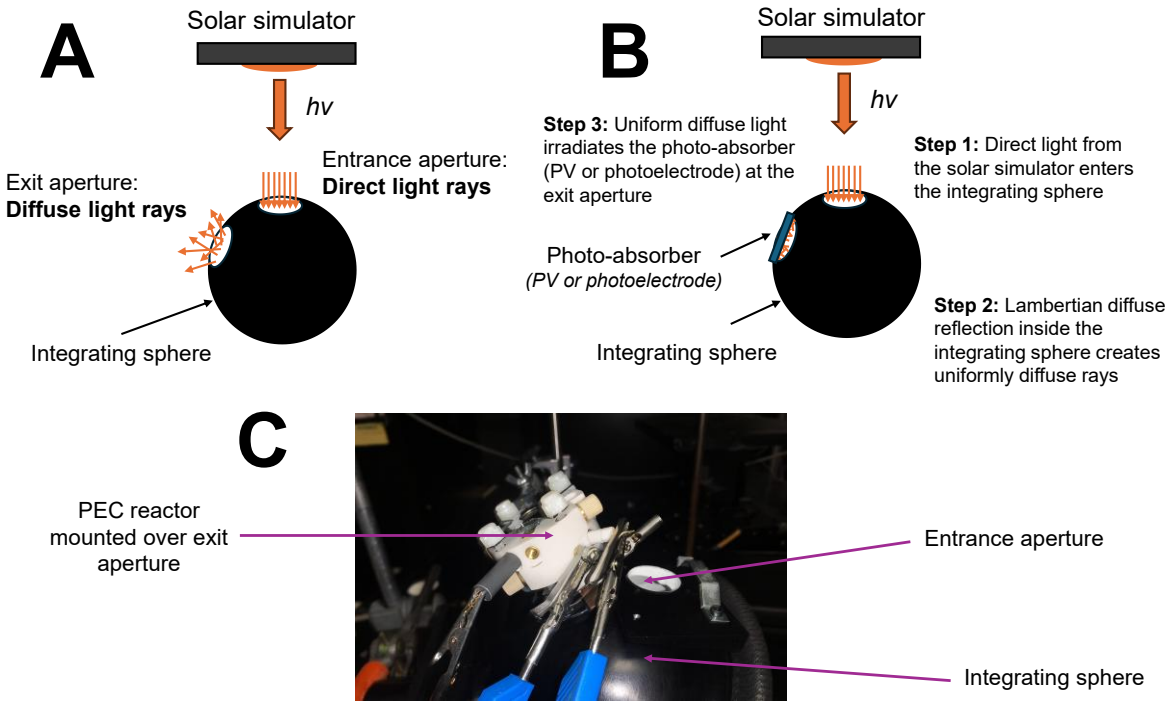

Figure 1. *(A, B) Schematic illustrations of the integrating sphere based diffuse light characterisation method. (C) Example of the path of a light ray inside the integrating sphere. (D) Schematic of the PEC reactor mounted over the exit aperture during characterisation and (E) corresponding photograph.*

A flat 1 cm^2 monocrystalline Si reference cell placed directly at the exit aperture was used to compare the total irradiance leaving the integrating sphere with the total irradiance at the entrance aperture (calibrated at 1 sun, 1000 W m^{-2}), to quantify the cumulative losses of internal reflections. Spectral measurements using the optical fibre-based spectrophotometers were not possible at the exit aperture, as their narrow angular acceptance prevents them from capturing the broad range of angles present under diffuse-like illumination. A planar surface integrates incoming light over the full angular distribution and therefore provides a representative measure of the effective irradiance experienced by the photo-absorbers at the exit aperture.

Electrochemical measurements were made using an Autolab PGStat302N potentiostat (Metrohm), interfaced with Nova 2.1 software. Photoelectrochemical measurements were conducted in a Redox.me PTFE PECF cell (adapted for quiescent measurements), equipped

with a Pt coil auxiliary electrode (Redox.me) and a refillable Ag/AgCl reference electrode (sat. KCl, 0.197 V_{SHE} at 25 °C, Redox.me). The electrolyte used was a 1 M sodium borate buffer (1 M H_3BO_3, ~17 g L^{-1} NaOH titrated to pH 9.3), prepared with Milli-Q-water (Millipore Corp., 18.2 MΩ cm at 25 °C). This electrolyte selection was informed by previous experiments in which analogous WO_3|$BiVO_4$|NiFeOOH photoanodes exhibited the highest stability in borate buffer [13]. The exposed (photoactive) area of the photoanodes in the PEC cell was 0.8 cm^2. The photoanodes were characterised under continuous illumination, at the entrance aperture (facing the solar simulator) and at the exit aperture (facing the inside of the integrating sphere), by linear sweep voltammetry from the open circuit voltage to 1.0 $V_{Ag/AgCl}$ (scan rate 10 mV s^{-1}).

WO_3|$BiVO_4$|NiFeOOH electrodes were fabricated on TEC-15 FTO substrates (Pilkington, UK) using aerosol-assisted chemical vapour deposition (AA-CVD) procedures previously established by the authors [13,14]. The CVD reaction was carried out using a purpose-built reactor (shown in **Figure S4**). UV-vis (shown in **Figure S5**) and XRD (shown in **Figure S6**) analysis showed that the materials fabricated in this work exhibited optical and structural properties consistent with those reported previously, indicating that the electrodes are representative of our established WO_3|$BiVO_4$|NiFeOOH photoanode structure.

For concentrated light experiments, a crossed-compound parabolic concentrator (CCPC) optic was mounted onto the surface of the photoanode using an epoxy resin, as shown in **Figure 2a**. The CCPC had angular acceptance of ± 30° and achieved an optical concentration ratio of 2.9. Ray-tracing diagrams of a CCPC optic at selected angles between 0° and 50° are shown in **Figure 2b** [15].

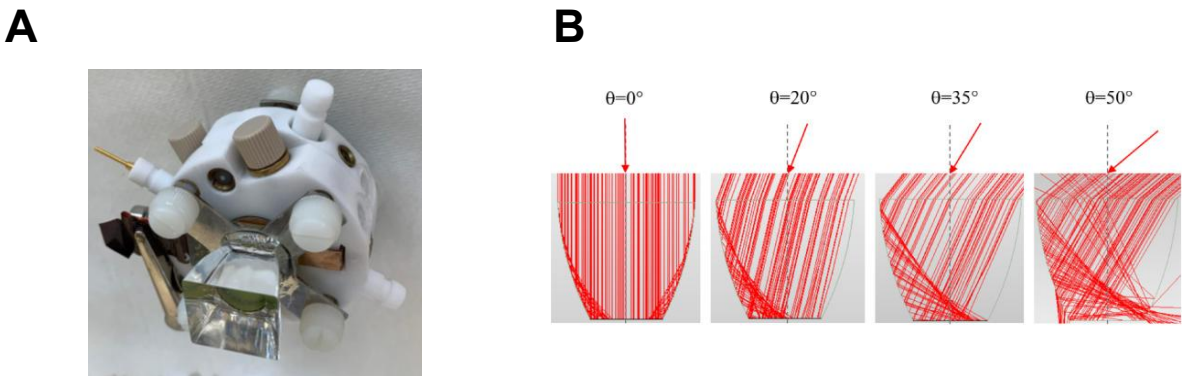

Figure 2. (A) *Photograph of the CCPC-integrated PEC reactor.* ***(B)*** *Ray-tracing diagrams of a CCPC optic at selected angles between 0° and 50°, reproduced under the terms of the Creative Commons CC-BY 4.0 licence. Copyright 2023, X. Li et al. [15]*

Results & discussion

Quantification of optical losses in the quasi-diffuse illumination module

To enable the comparison of photoelectrochemical performance under irradiation of quasi-diffuse light with the predominately collimated, direct illumination provided by the solar simulator, it was necessary to quantify the optical losses within the integrating sphere arising from internal reflectance and port losses through the entrance aperture. The effective irradiance delivered at the exit aperture of the integrating sphere was quantified by comparing the short circuit current of a 1 cm^2 monocrystalline solar cell with a linear current-irradiance response characteristic of silicon photovoltaic devices under standard operating conditions. As summarised in **Table 1**, reflectance losses and port losses resulted in a 76.3 % reduction in the relative irradiance at the exit aperture (to 0.237) compared with the entrance aperture. Reporting the relative irradiance at the exit aperture will enable reproducible comparison between different implementations of this method. Subsequent current densities were scaled according to the relative irradiances in **Table 1**, to enable the comparison between direct and diffuse illumination conditions, with raw (unscaled) data provided in the supplementary information as appropriate.

***Table 1**. Relative irradiance at the entrance and exit aperture of the integrating sphere for a calibrated irradiance at the entrance aperture of 1000 W m^{-2} (i.e. 1 sun, AM 1.5 G spectrum). The relative irradiance was quantified by comparing the short circuit current of a 1 cm^2 monocrystalline solar cell positioned at the entrance and exit apertures. The locations of the entrance and exit apertures on the integrating sphere can be seen in Figure 1a.*

Illumination condition	Measurement plane	I_{sc} (mA)	Relative irradiance
Direct (collimated)	Entrance aperture	44.7	1.000
Quasi-diffuse	Exit aperture	10.6	0.237

Effect of quasi-diffuse irradiation on photoelectrochemical performance

To examine the effect of the quasi-diffuse irradiation from the integrating sphere on photoelectrochemical performance, current densities generated during linear sweep voltammetry under predominantly collimated, direct illumination and quasi-diffuse irradiation (at the integrating sphere exit aperture) were compared. These data are illustrated in **Figure 3**. The irradiance under direct illumination (at the entrance aperture of the integrating sphere) was 1000 W m^{-2} (1 sun, AM 1.5G). The effective irradiance under quasi-diffuse illumination at the exit aperture was 237 W m^{-2} and current densities in **Figure 3** have therefore been scaled to one-sun equivalent. The corresponding as-measured LSV curve from the measurement at the exit aperture is shown in **Figure S7**.

The comparable performance under direct and quasi-diffuse irradiation after intensity scaling to one sun equivalent indicates that the photoelectrochemical performance of a planar photo-absorber (without concentrating optics) is primarily governed by the total photon flux, rather than the angular distribution. This further suggests that under non-clear sky conditions the primary driver of decreased photoelectrochemical performance is the reduction in irradiance associated with cloud attenuation and atmospheric scattering or absorption [16].

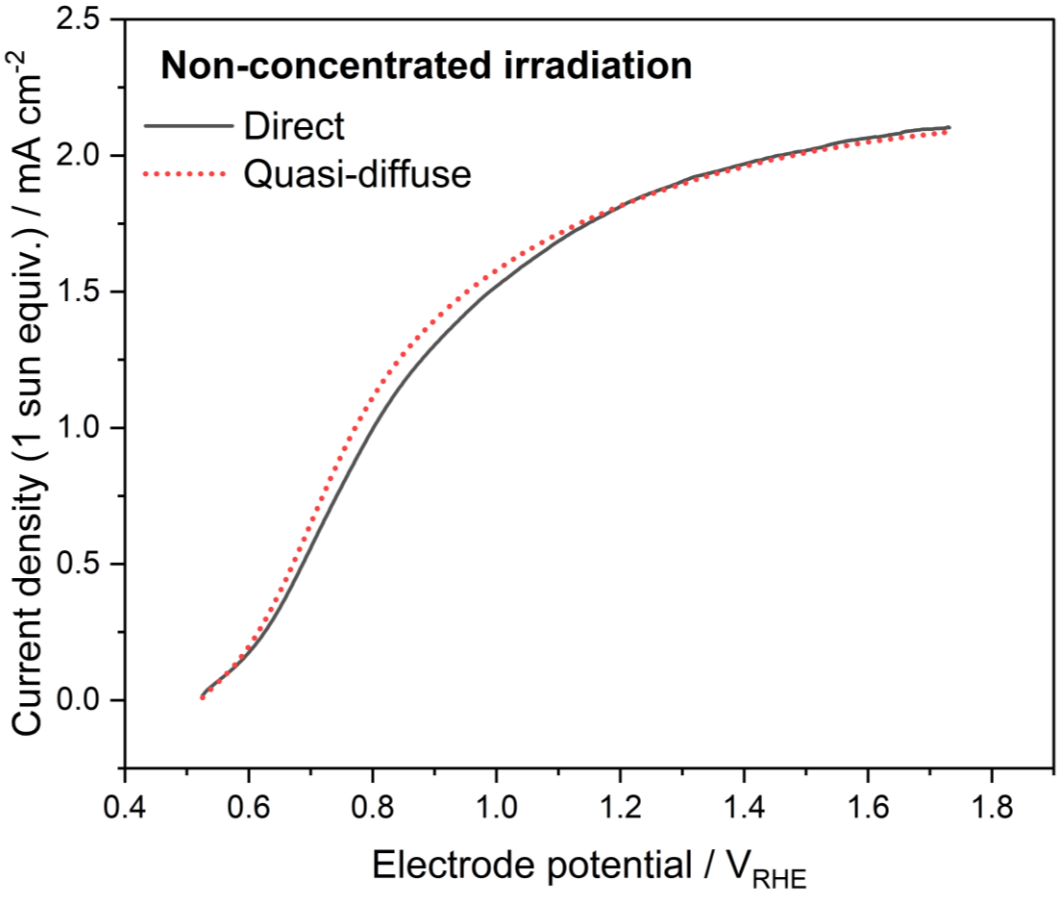

Figure 3. *Effect of direct illumination and quasi-diffuse illumination (delivered at the exit aperture of the integrating sphere) on the photoelectrochemical performance of WO_3\|$BiVO_4$\|NiFeOOH. To enable comparison of direct and quasi-diffuse performance, the quasi-diffuse current density was normalised to one sun equivalent, with the original curves available in Figure S7.*

Although demonstrated here for a WO_3\|$BiVO_4$\|NiFeOOH photoanode, a similar behaviour is expected for the characterisation of other planar photoelectrodes and conventional flat-plate PV cells. This irradiance-dominated behaviour is characteristic of planar photo-absorbers. Device architectures that incorporate concentrators or other optics are expected to respond differently, as will be demonstrated in the following subsection.

Quasi-diffuse light concentration using a non-imaging CCPC optic

In contrast to imaging optics, which offer high optical concentration ratios but have very narrow angular tolerances, non-imaging optics can enhance effective irradiance whilst retaining wide

acceptance angles [10,11]. CCPCs have a design optical concentration ratio of 3.6 and a half acceptance angle of ± 30° [15,17]. When integrated with a PEC reactor, the benefits of increased effective irradiation can be realised, even with diffuse or indirect light, whilst avoiding detrimental device heating that occurs under higher solar concentration [8].

Due to their very narrow angular acceptance, the efficiency of imaging optics is severely limited by non-clear sky conditions when there is a greater degree of diffuse irradiance and when the incident angle distribution broadens at low solar elevation. Non-imaging optics, such as CCPCs, continue to deliver a concentration benefit under these conditions, enabling device operation beyond ideal clear-sky illumination. This makes wide-angle non-imaging concentrators a realistic route to improved PEC performance in diffuse-rich or low elevation environments.

Figure 4 illustrates a comparison of the effect of direct and diffuse CCPC light concentration on PEC performance. As with the non-concentrated light example, the effective irradiance under quasi-diffuse illumination at the exit aperture was 237 W m^{-2} and current densities were therefore scaled to one-sun (1000 W m^{-2}) equivalent. The corresponding LSV curve for the CCPC condition under quasi-diffuse irradiation is shown in **Figure S8**.

Under quasi-diffuse irradiance, the CCPC retains a useful, acceptance angle limited concentration effect, with the normalised current density approximately 30 % of the value under direct light. This demonstrates that wide-angle non-imaging optics can retain a measurable concentration benefit under diffuse-rich illumination. In contrast, imaging optics, such as convex lenses, have very narrow angular acceptance (typically to the order of 1 or 2°), and under uniformly diffuse irradiation cannot form a focused spot. For small focal receivers (typical in such systems) this results in negligible irradiance delivered to the photo-absorber and thus negligible photocurrent.

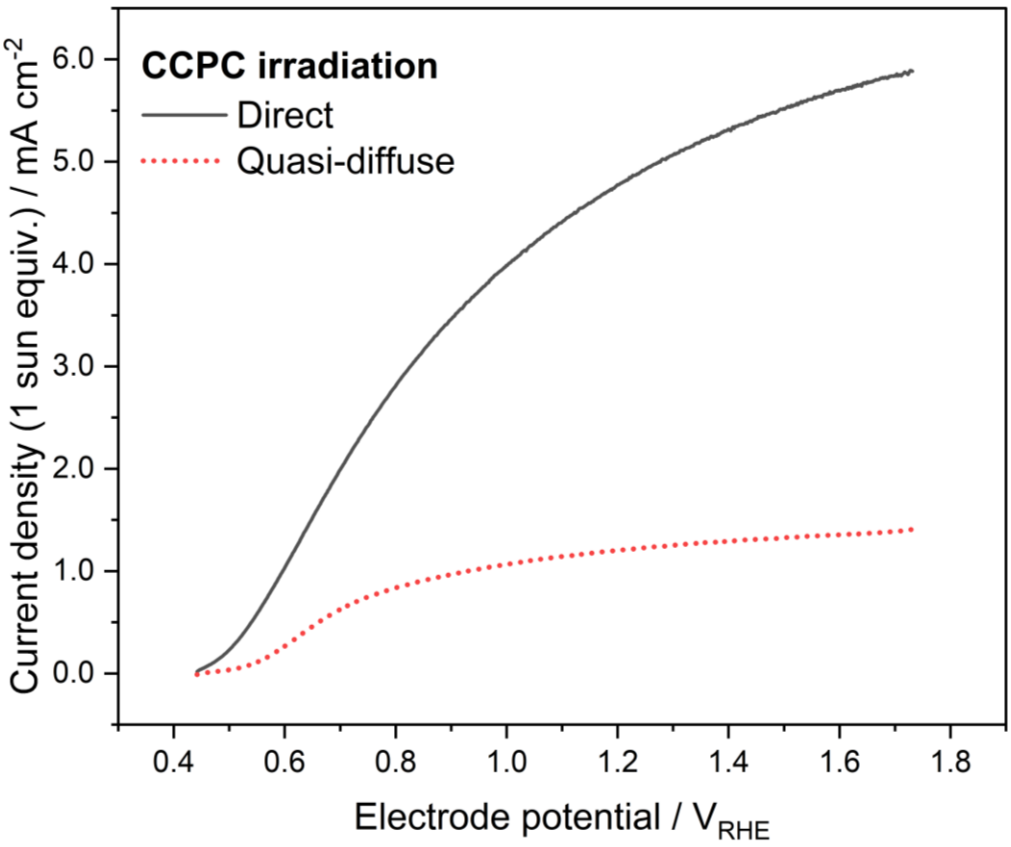

***Figure 4**. Effect of direct illumination and quasi-diffuse illumination (delivered at the exit aperture of the integrating sphere) on the photoelectrochemical performance of WO_3|$BiVO_4$|NiFeOOH coupled with a CCPC optic. To enable comparison of direct and quasi-diffuse performance, quasi-diffuse current density was normalised to one sun equivalent, with the original curve available in Figure S8.*

Implications for evaluating devices and optics under diffuse light conditions

The non-concentrated WO_3|$BiVO_4$|NiFeOOH PEC demonstration showed that, at matched one sun equivalent irradiance, the PEC performance was comparable under both direct and quasi-diffuse irradiance, indicating an irradiance-dominated response. This flux-dominated behaviour is expected to extend to other photo-absorbers with similar, planar geometries, consistent with flat-plate PV studies and PEC benchmarking protocols, which treat total irradiance as the primary driver of performance [18,19].

In contrast, our use of a CCPC showed that optical and device architectures can have a markedly different effect on device performance, in this case driven by the wide acceptance angle of the CCPC optic [15,20]. This method can therefore enable ray tracing and other optical model validation in a straightforward, reproducible way, without the need for complex procedures or the use of facilities that typical solar energy research groups would not have access to [8,12,21]. The ≈ 30 % retention in photocurrent with the CCPC is consistent with non-imaging theory and modelling, which shows that wide-angle accepting solar optics can concentrate part of the diffuse component [15].

Whilst these insights could also be gathered from outdoor field trials, these can be complex to organise, and experiments are subject to unpredictable weather conditions and temperature fluctuations [8,12]. Furthermore, variation in weather conditions, even in the same testing locations, can lead to different results, making it more difficult to compare performance across different research teams. This work provides a framework to complement existing standards for testing solar PV and PEC devices, which are typically focused on AM 1.5 G one-sun characterisation conditions, by adding a controllable quasi-diffuse test which is more representative of climates with high proportions of diffuse irradiance [19,22]. Used alongside AM 1.5 G characterisation, this method enables a clearer picture of the performance of different photo-absorbers and optical architectures across a wider range of conditions, in a reproducible and controllable way.

Conclusions

In this work, we developed a reproducible method for characterising photo-absorbing materials under diffuse irradiance. This involved coupling a solar simulator with an integrating sphere typically used in UV-vis-NIR spectrophotometry applications. By directing the primarily collimated light of the solar simulator into the integrating sphere, we were able to irradiate the photo-absorber with quasi-diffuse “cloud-like” light scattering at the exit aperture. To demonstrate the use of this technique, we contrasted two device architectures, using a

WO_3|$BiVO_4$|NiFeOOH photoanode-based PEC reactor. First, we compared the effects of direct and diffuse light on the planar photoanode surface. At normalised solar flux, the performance of the direct and quasi-diffuse conditions was very similar, indicating an irradiance-dominated response for planar geometries. Next, we coupled the PEC reactor with a crossed-compound parabolic concentrator (CCPC), a non-imaging optic with wide-angle acceptance. In this case study, there was a ≈30 % retention in photocurrent in the diffuse condition, showing the effect of the angular acceptance of the CCPC optic.

This study provides a framework for diffuse light testing that can complement existing standards which are primarily centred around direct light, relatively high irradiance conditions, but may not necessarily represent realistic operating conditions. This method enables simple, rapid validation of ray tracing and other optical models and provides a practical way to benchmark photo-absorber performance under a wider range of illumination conditions than are typically characterised in the laboratory, without the need for dedicated field trials.

Author contributions

George H. Creasey: methodology, investigation, formal analysis, writing – original draft, writing – review and editing, visualisation, project administration, funding acquisition. **Andreas Kafizas:** resources, writing – review and editing, supervision. **Anna Hankin:** resources, writing - review and editing, supervision, funding acquisition. **Katie Shanks:** conceptualisation, methodology, investigation, resources, writing – review and editing, supervision.

Conflicts of interest

There are no conflicts of interest to declare.

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