Roadmap towards solar fuel synthesis at the water interface of liposome membranes

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Supplementary information

Table S1. Experimental techniques for the preparation, characterisation, and study of photocatalytic self-assembled membranes.

Experimental techniques	Information obtained	Reference(s)
Nuclear Magnetic Resonance (NMR)	– Nature and purity of membrane-embedded components – Stability of liposomes and membrane-embedded components – Photoproduct analysis and quantification (e.g. formate from CO_2 reduction)* – Position and diffusion of membrane-embedded components (Magic Angle Spinning NMR, MAS NMR)	1–5
Fourier Transform / Attenuated Total Reflectance Infrared Spectroscopy (FT/ATR-IR)	– Confirm presence/immobilisation of membrane components* – Lipid membrane phase transitions	6–9
Mass Spectrometry (MS, native and LC-MS, HPLC-MS)	– Characterisation of the membrane-immobilised components – Determination of reaction products and decomposition by-products of the membrane (lipid degradation)	10
Cryo Transmission Electron Microscopy (TEM), Cryo Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX)	– Morphology and size of liposomes (cryo TEM, SEM) – Characterisation and quantification of metal-based membrane components (EDX) – Stability studies	11–13
Electron Paramagnetic Resonance (EPR)	– Determination of the physical properties (phase transition, fluidity) of a membrane – Determination of the pH at membranes – Study of oxygen transport in membranes (spin labels) – Study of oxidation of lipids in membranes – Characterisation of paramagnetic membrane-embedded components	14–16
X-ray Photoelectron Spectroscopy (XPS)	– Characterisation and quantification of metal-based membrane components*	17,18
UV-Vis spectroscopy	– Characterisation of electronic absorption properties of membrane-immobilised components – Characterisation of the filter effect – Stability of liposomes and membrane-embedded components	12,19–32
Fluorescence/emission spectroscopy	– Characterisation of excited state energy of membrane-embedded components – Stability of liposomes and membrane-embedded components – Transition temperature of heterogenous mixture of lipids/molecules	6,10,11,21,22,24,26–28,32–35
Time-resolved absorption or emission spectroscopy	– Determination of excited state lifetime of membrane-embedded photoactive components – Analysis of the binding of immobilised components within the liposome assembly – Nature and kinetics of reactive intermediate species during photocatalysis (mechanism)	11,20,22,24,28,30,32,33,36,37
Single-molecule fluorescence microscopy	– Determination of the lipid lateral diffusion in membranes	38
Differential Scanning Calorimetry (DSC) and fluorescence anisotropy	– Determination of the membrane transition temperature	11,39
Dynamic and static light scattering	– Hydrodynamic diameter of liposomes – Surface charge of liposomes / zeta (ζ) potential – Stability of liposomes and membrane-embedded components	6,12,19,23,25–29,32,34,40,41
Nanoparticle tracking analysis	– Concentration and size distribution of liposomes	19
Atomic Force Microscopy (AFM)	– Analysis of the surface of liposomes	13,42

Inductively Coupled Plasma - Mass Spectrometry (ICP-MS), Atomic Emission Spectroscopy (ICP-AES), Optical Emission Spectroscopy (ICP-OES)	– Quantification of metal content (synthetic molecules and enzymes)	23,43,44
Confocal microscopy	– Assessment of membrane-embedding of luminescent components (only in giant unilamellar vesicles)	1,13,32,34,35
Extrusion kit (syringes, polycarbonate extrusion filters, etc.)	– Prepare monodispersed liposome solutions	6,10,11,21–23,25,29,32–34,40,41,45
Cyclic voltammetry	– Electrochemical characterisation of lipid membrane-embedded components (quantification, stability, activity)*	8,37,46–49
Sonicator, ultrasonicator	– Preparation of small unilamellar liposomes	6,11,12,20,24,26–28,45
Rotary evaporator and liquid N_2 Dewar	– Preparation of liposomes (before hydration/self-assembly) and storage	12,21–23,25,32,34,45
Column chromatography (e.g. size exclusion gel filtration, affinity columns, desalting column, etc.)	– Purification of proteo-liposomes or liposomes after self-assembly with newly immobilised synthetic components	6,10,19,20,22,23,25,26,32,34,41,45
Light source (e.g. LED 450 nm for $[Ru(bpy)_3]^{2+}$; sunlight simulator $\lambda > 400$ nm, Xe lamp, 100 mW cm^{-2}, AM 1.5G)	– Light irradiation for photocatalytic experiments	12,21,22,24–28,30–32,35,37
Fluorescence O_2 probe or Clarke electrode	– Quantification of O_2	12
Gas Chromatography (GC)	– Product analyses and quantification (gaseous product e.g. H_2, O_2, CO)	12,26–28,30,31
Ion Chromatography (IC)	– Product analyses and quantification (charged ions e.g. formate)*	5
Osmometer	– Determination of osmolarity of buffer solution containing components (for preparing asymmetric liposomes)	12,25

* Universal method, mainly used in homogeneous or heterogeneous systems.

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