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Data article

Pressure-dependent inverse bicontinuous cubic phase formation in a phosphatidylinositol 4-phosphate/phosphatidylcholine system

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ABSTRACT

In this paper, we report the inositide-driven formation of an inverse bicontinuous cubic phase with space group Ia3d (Q_{II}^{G}, gyroid phase). The system under study consisted of distearoylphosphatidylinositol 4-phosphate (DSPIP) and dioleoylphosphatidylcholine at a molar ratio of 1:49, with a physiological concentration of magnesium ions at *p*H 7·4. The behaviour of the system was monitored as a function of temperature and pressure. The formation of the phase with Ia3d geometry was recorded repeatably at high pressure, and occurred more readily at higher temperatures. We conclude that the Ia3d phase formed is a thermodynamically stable structure, and that DSPIP is a potent source of membrane curvature that can drive the formation of mesophases with both 2- and 3D geometry.

Abbreviations: DOPC, Dioleoylphosphatidylcholine; DSPIP, distearoylphosphatidylinositol 4-phosphate; H_{II}, Inverse hexagonal; MLV, Multi-lamellar vesicle; PI-4-*P*, Phosphatidylinositol 4-phosphate.

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Specifications table	
Subject area	Chemical biology, Physical chemistry
Compounds	Distearoylphosphatidylinositol 4-phosphate, dioleoylphosphatidylcholine
Data category	Physicochemical, biophysical
Data acquisition format	X-ray diffraction
Data type	Analysed
Procedure	Temperature and pressure scans of hydrated lipid mixture as a model for a biological membrane
Data accessibility	Data is in this article.

1. Rationale

Research interest in lipid aggregations with cubic geometry has increased in recent years, owing to suggestions that swollen cubic phases are a model for the membrane division events observed *in vivo*. Such events include endocytosis [1–3], vesicle formation [4,5] and vesicle trafficking [6]. Work on model lipid systems has provided evidence for induction of vesicle budding with changes in buffer [7] and for cubic phases that are hydration-dependent [8] and tuneable with respect to pressure [[9], [10]]. The appearance in model systems of lipid aggregations of dimensions and geometry similar to those in nature, but with non-natural lipid mixtures, led us to the hypothesis that naturally-occurring lipid isoforms of certain lipids may effect the same shifts in model systems under application of hydrostatic pressure.

Recent reports have provided evidence that the inositide group of phospholipids (also known as phosphatidylinositol or PI lipids) can drive the formation of curved lipid aggregations [11,12]. These physical observations may be very significant in interpreting cellular levels of inositides during processes involving the generation of membrane curvature, such as cytokinesis in HeLa cells [13] and endocytotic processes in pollen [14]. The evidence surrounding the physical behaviour of inositides has led to suggestions [15] that their physical role(s) may be as significant as their well-established signalling ones [16–18]. This body of work led us to explore the phase behaviour of a typically lamellar system that had been doped with a PI-4-*P*.

2. Procedure

DOPC was obtained from Avanti Polar Lipids (Alabaster, AL) and used without further purification. All salts, solvents and buffers were obtained from Sigma Aldrich (St. Louis, MO). Racemic DSPIP was prepared in our laboratory [19,20], as its bis-ammonium salt.

2.1. Sample preparation and equilibration

All samples were prepared by dissolving the lipids in a mixture of solvents ($CHCl_3$: CH_3OH : H_2O; 7:2:0·1). These were then mixed in the required molar ratios, the resulting mixtures were then frozen using liquid nitrogen and freeze-dried over a 48 h period to ensure that all solvent was removed from the sample. Approximately 3 mg of lipid was added to an X-ray capillary (Gulmay Medical Ltd, UK, 1.5 mm) and hydrated with 66% wt/wt buffer. The buffer used for sample hydration contained $MgCl_2$ (5 mM) and Tris-base (20 mM) adjusted to *p*H 7·4 at 25 °C. The glass capillaries were flame sealed, and then sealed with silicone sealant (Dow Corning Corp) to maintain a fixed hydration level. The uncertainty in the concentration $\Delta c/c$ has been estimated to be 1–2wt%. The sample was allowed to equilibrate for 7 days at 25 °C before use. The weight of the sealed capillary before and after each X-ray diffraction experiment was noted and compared to the initial tare, with no water loss observed. Samples were equilibrated in the synchrotron for 15 min at each temperature before acquisition, and for 10 min before acquisition at each pressure, in agreement with previous studies [11,12].

2.2. X-ray instrumentation

Synchrotron small-angle X-ray scattering (SAXS) measurements were carried out at the ID02 high brilliance beamline at the European Synchrotron Radiation Facility (ESRF) in Grenoble (France),