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Reorganization of lipid nanocapsules at air–water interface Part 2. Properties of the formed surface film

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Abstract

The state, electrical and dilatational rheological properties of surface films formed at air–water interface from lipid nanocapsules (LNC) with various compositions as well as model monolayers formed by the LNC constituents—Labrafac®, Solutol® and Lipoid® are investigated. These nanocapsules constitute potential drug delivery systems where lypophilic drug will be loaded in their core. The study of the model Labrafac®/Solutol® (Lab/Sol) mixed monolayers shows behavior close to the ideal. Small negative deviations in the mean molecular areas a and dipole moments μ are observed. All studied monolayers have elastic behavior during the small continuous compressions. The comparison between the properties of surface films formed from LNC with those of the model monolayers confirms the idea developed in the kinetic study [I. Minkov, Tz. Ivanova, I. Panaiotov, J. Proust, P. Saulnier, Reorganization of lipid nanocapsules at air–water interface: 1. Kinetics of surface film formation, J. Colloids Surf. B: Biointerfaces, submited for publication.] that the surface films formed after a rapid disaggregation of the unstable nanocapsule fraction (LNC I) contains mainly Labrafac and Solutol. The Labrafac molar part (x_{Lab}) in the formed Lab/Sol mixed layer is established.
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Keywords: Lipid nanocapsules; Spreading; Surface film; Labrafac

1. Introduction

During these last decades, the formulation at the nanometric scale of drug delivery systems is a strong challenge of the pharmaceutical field. The goal is to be able to release in vivo, a sufficient amount of drug in a specific place. Synthetic nanoparticles need to respect numerous specifications in order to constitute operant vectors [2].

Mainly these particles have to protect the drug against inactivation but also to protect healthy organs from possible toxicity of this drug. One has to avoid the immune system as well as the protecting action of all the biological barriers. Finally, one has to target sufficient amount of active drug that will be released by diffusion of after destruction of the particle. Fortunately, natural vectors exist (lipoproteins, virus, etc.) but their reconstitution or manipulation in a laboratory can be strongly problematic [3].

In this way, the challenge is to formulate nanometric synthetic particles with demonstrated biomimetic properties. A novel class lipid nanocapsules (LNC), biocompatible, nontoxic and remaining stable in the bulk of the aqueous suspensions was recently proposed [4,5]. Nevertheless, a direct study at the air/water interface of the behavior the LNC constitute powerful tool in the characterization of the structure and the stability of these systems. In the first part of these series [1], the mechanisms of loss of mechanical stability of the lipid nanocapsules at air/water interface were investigated. At air–water interface the LNC undergo destabilization and disaggregation, leading to formation of a surface film, which contains the LNC constituents Labrafac®, Solutol® and Lipoid®. One has to note that Polyethylene Groups of Solutol® confer important stealthy properties that were demonstrated for this kind of particles [6].

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The aim of this paper is to study the state, electrical and rheological properties of the formed surface films. Their behavior is compared with the properties of model systems—monolayers composed by the LNC constituents (Labrafac®, Solutol® and Lipoid®) obtained by the classical procedure from volatile solvents.

The rheological properties are very sensitive to the state and the organization of two-dimensional (2D) monolayers. The dynamic response of a surface film to a dilatational (compressional) mechanical stress for characteristic times in the range 1–100 s is investigated by the method of continuous compression (or expansion) [7–10].

2. Materials and methods

2.1. Materials

The complete description of the used materials, Labrafac®, Lipoid®, Solutol® and NaCl is presented in the first part of this series—Kinetics of surface film formation [1].

2.2. Methods

The monolayers were formed by spreading the specified amounts of the solutions over the available area of the trough. The spreading was performed by using Exmire microsyring. The measurements were performed at room temperature $-22 \pm 2\,^{\circ}$C. The surface pressure (π) was measured by using a KSV-2200 (Finland) surface balance, equipped with a platinum plate. The surface potential (ΔV) was measured simultaneously by using a gold-coated ^{241}Am ionizing electrode, a reference electrode and a KP 511 (Kriona, Bulgaria) electrometer, connected to a PC provided with user software for real-time data measurements. The reproducibility of the initial surface potential value was ± 15 mV. When the air–water surface potential became constant, a spreading of the monolayer was performed. After an equilibration period of about 5–10 min, the surface potential became constant and the following kinds of experiments were performed:

1. Surface pressure – area and surface potential – area isotherms were obtained after spreading on double distilled water of various quantities M_i of three different LNC aqueous suspensions. The isotherms of chloroform solutions of the pure components (Labrafac, Solutol and Lipoid) and their mixtures were also obtained. Depending on the initially used molar part of Lipoid (x_{Lip}) during the preparation, the different LNC suspensions are called: sample 1 (without Lipoid), sample 2 with a molar part of Lipoid $x_{\mathrm{Lip}} = 0.3$ and sample 3 with $x_{\mathrm{Lip}} = 0.9$. Detailed information of the initially used amounts for each component and the preparation procedure is available in the first part of the series [1].

   The monolayers were continuously compressed with a constant velocity $U_{\mathrm{b}} = 50\,\mathrm{cm^2\,min^{-1}}$ at the available area of the teflon trough ($350\,\mathrm{cm^2}$).
2. The rheological dilatational properties of the monolayers were studied by measuring the surface pressure variations with time along the monolayer during and after a small continuous compression. The method described in Refs. [7–10] consists of compressing the monolayer by means of a barrier moving with a constant velocity U_{b} (Fig. 1A and B).

   As a result of the surface density gradient, caused by continuous local surface pressure perturbation, a simultaneous motion of the monolayer and the liquid substrate occurs. The surface motion is practically dilatational and can be detected (as shown in Fig. 1A) by measuring with a Wilhelmy plate, the changes in the local surface pressure at points x_1, x_2 and x_3. A complete description of the simultaneous motion of the monolayer and liquid substrate was made [7–10]. In the absence of intrinsic surface dilatational viscosity or other relaxation processes (solubilisation, 2D reorganization, collapse, etc.) during the compression, the rheological behavior of an insoluble monolayer is elastic and can be described by the Hook's

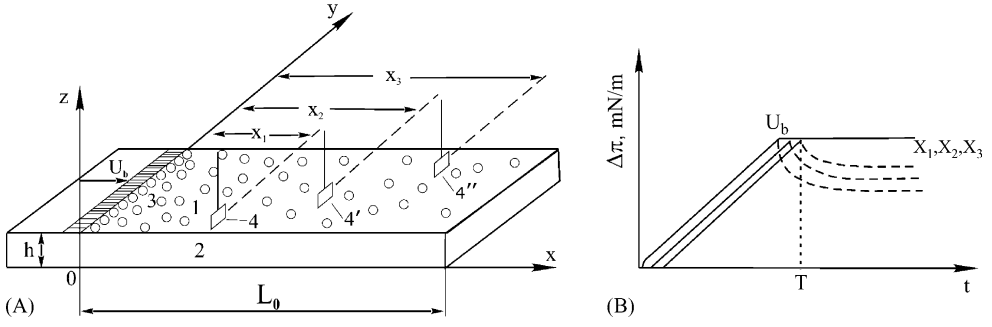

Fig. 1. (Panel A) Langmuir trough: 1, monolayer with length L_0; 2, subphase with depth h; 3, barrier, which moves with a constant rate U_{b} ($U_{\mathrm{b1}} = 180\,\mathrm{cm^2\,min^{-1}}$ and $U_{\mathrm{b2}} = 10\,\mathrm{cm^2\,min^{-1}}$); 4, 4′ and 4″, Wilhelmy plates located at tree different distances x_1, x_2 and x_3, respectively. (Panel B) Variation of a surface pressure $\Delta\pi$ during ($t < T$) and after ($t > T$) compressions with velocity U_{b}. The full lines correspond to elastic monolayer. The dashed lines show the behavior of a monolayers witch are visco-elastic after the end of the compression ($t > T$).

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