

# Mössbauer study of C18N/Fe Langmuir-Blodgett layers

Erno Kuzmann · Judit Telegdi · Zoltán Németh ·  
Attila Vértes · Lajos Nyikos

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**Abstract** Langmuir-Blodgett (LB) films of octadecanoyl hydroxamic acid (C18N) complexed with  $\text{Fe}^{3+}$  ions have been prepared at various subphase pH values. The LB films consisting of different number of layers were investigated by  $^{57}\text{Fe}$  conversion electron Mössbauer spectroscopy (CEM) at room temperature. The CEM detector contained a piece of  $\alpha$ -iron, enriched with  $^{57}\text{Fe}$ , using as an internal standard. The Mössbauer pattern of the C18N/Fe LB films is a doublet with parameters  $\delta = 0.35$  mm/s and  $\Delta = 0.74$  mm/s. A gradual increase of the relative occurrence of the doublet compared to the sextet of the internal standard was observed with the increasing number of layers, indicating the nearly uniform distribution of Fe among the LB layers.

**Keywords** Langmuir-Blodgett layer ·  $^{57}\text{Fe}$  CEM Mössbauer spectroscopy · Internal etalon · Octadecanoyl hydroxamic acid

## 1 Introduction

Amphiphilic molecules with small ionic head group and long hydrophobic chains are suitable for preparation of ultra thin, dense, protective Langmuir-Blodgett (LB) layers. These LB films on metal surfaces like copper could be applied as potential anticorrosive and bacterial adhesion preventing coatings [1]. In order to produce the most compact LB layer, the structure and behavior of amphiphiles in the molecular layer at the air-water interface should be very precisely monitored as the physical

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E. Kuzmann · Z. Németh (✉) · A. Vértes  
Institute of Chemistry, Eötvös Loránd University, Budapest, Hungary  
e-mail: hentes@chem.elte.hu

J. Telegdi · L. Nyikos  
Institute of Nanochemistry and Catalysis, Chemical Research Center,  
HAS, Budapest, Hungary

and chemical parameters of the subphase and the Langmuir monolayers influence the quality of LB films deposited onto solid supports [2, 3]. In order to incorporate counter ions into the LB film, salt of  $\text{Fe}^{3+}$  ions is dissolved in the aqueous subphase. At suitable pH value which influences the ionization of amphiphiles, the  $\text{Fe}^{3+}$  ions could form complex with the organic molecule. The aim of the present work was to study the incorporation and distribution of iron in the LB films of octadecanoyl hydroxamic acid (C18N) at various subphase pH values.

## 2 Experimental

6.5 mg Octadecanoyl hydroxamic acid (C18N) synthesized in our laboratory was dissolved in 10 ml of chloroform and used as stock solution. An aqueous solution of  $\text{Fe}^{3+}$  ions (chloride salt of  $\text{Fe}^{3+}$  ions at  $5 \times 10^{-5}$  M concentration) was used as a subphase at various pH values. For the Mössbauer study 90%  $^{57}\text{Fe}$  enriched iron isotope was used. The pH was adjusted to the desired value by adding dilute solutions of NaOH or HCl. A proper amount of the stock solution was spread onto the aqueous subphase in a Langmuir trough (NIMA Technology Ltd., 611D) by micro syringe. After complete evaporation of the organic solvent (10 min) the compression was initiated at a speed of  $200 \text{ cm}^2 \text{ min}^{-1}$ . Molecular area isotherms were obtained under continuous compression in the LB trough. Surface pressures were measured with a Wilhelmy plate balance.

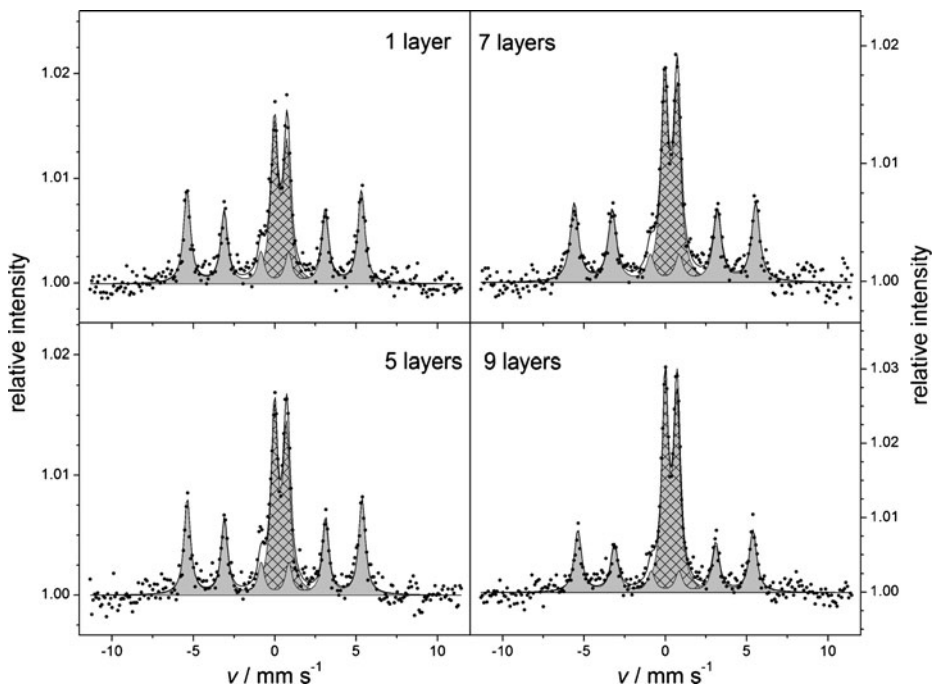
$^{57}\text{Fe}$  Mössbauer spectra of LB samples were recorded in reflection geometry with a conventional Mössbauer spectrometer (WISSEL) working in constant acceleration mode of conversion electron Mössbauer measurement using a RANGER-type CEM detector at room temperature. The CEM detector contained a piece of  $\alpha$ -iron, enriched with  $^{57}\text{Fe}$ , using as an internal standard. The  $\gamma$ -rays were provided by a  $3 \times 10^9 \text{ Bq } ^{57}\text{Co/Rh}$  source. Isomer shifts are given relative to  $\alpha$ -iron. The Mössbauer spectra were analyzed by least-square fitting of Lorentzian lines by the help of the MOSSWINN code [4].

## 3 Results and discussion

The typical room temperature CEM spectrum of the C18N/Fe LB layers is a paramagnetic doublet with average isomer shift,  $\delta = 0.35 \text{ mm/s}$  and quadrupole splitting,  $\Delta = 0.74 \text{ mm/s}$ . These parameters reflect the occurrence of iron in valence state of  $\text{Fe}^{3+}$  in the LB layer. This result is confirmed by previous Mössbauer results, obtained also in transmission geometry, of LB layers [1].

Figure 1 shows room temperature CEM spectra of C18N/Fe LB films consisting of different number of layers. The envelop of the spectra was decomposed into a doublet and a sextet. The doublet corresponds to the iron microenvironment in the C18N/Fe LB layers. The sextet is the fingerprint of  $\alpha$ -iron belonging to the internal etalon placed previously in the CEM detector in order to serve as a standard for the determination of the relative area of the doublet component at different layer numbers.

We have found a significant increase in the relative area of the doublet with the increasing number of LB layers (Fig. 1), in comparison with the relative area of the



**Fig. 1**  $^{57}\text{Fe}$  CEM spectra of C18N/Fe Langmuir-Blodgett films consisting of different number of layers. The sextet is the fingerprint of  $\alpha\text{-Fe}$  used as an internal standard built in the CEM detector

$\alpha\text{-Fe}$  used as an internal standard. This result gives information about the distribution of iron among the individual LB layers. Based on the spectrum evaluations, the calculation of iron distribution showed that each LB layer contains iron and the distribution of iron is nearly uniform among the LB layers.

Our result indicates a nearly uniform incorporation of iron into the LB nanolayers of amphiphiles with hydroxamic head groups, resulting an enhanced anticorrosion effect of the LB film [1], via the formation of more compact hydrophobic region of the layers kept together mainly by van der Waals forces and by the interaction of the head groups.

#### 4 Conclusions

A nearly uniform distribution of iron in the Langmuir-Blodgett films of octadecanoyl hydroxamic acid (C18N) complexed with  $\text{Fe}^{3+}$  ions has been proven by the help of  $^{57}\text{Fe}$  conversion electron Mössbauer spectroscopy using  $^{57}\text{Fe}$  enriched  $\alpha\text{-iron}$  as internal etalon built in the CEMS detector. The result of the Mössbauer spectroscopy gives an explanation for the increased anticorrosion and antifouling activity of the LB layers even with a very low iron concentration.

**Acknowledgements** The research was supported by grants of the Hungarian Science Foundation (OTKA K68135, K100424 and NN84307)

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