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Langmuir films of dichroic dyes with fluorescent properties

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Abstract

Langmuir films of various dyes, namely derivatives of 3,4,9,10-perylenetetracarboxylic acid and 3,4,9-perylenetricarboxylic acid, derivatives of naphthalenebicarboxylic acid and derivatives of naphthoylenebenzimidazole were investigated. Surface pressure–mean molecular area isotherms were recorded from which the alignment of the molecules in a monomolecular layer at the air–water interface was determined. It was found that both the stability and the rigidity of the monolayer are strongly affected by the molecular structure of the side groups substituted on the skeleton of the molecule.

Keywords: Monolayer; Dichroic fluorescent dyes; Surface pressure–area isotherm

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1. Introduction

Since the 1970s when Kuhn et al. [1] published their stimulated work on energy transfer in multilayer systems there has been a long-standing interest in the utilization of the Langmuir–Blodgett (LB) technique [2–6] in molecular engineering. The LB technique is a unique method which offers the possibility of producing ultrathin, ordered layers from certain molecules or particles, the architecture of which can be manipulated readily. This permits optimization of a specific physical parameter of a material and, therefore, LB films are nowadays an integral component of molecular electronics [4]. Some years ago Stapff et al. [7] proposed to use the LB technique to obtain organic light emitting diodes (OLEDs). The basic requirements for the organic layer used in OLEDs are that it exhibits luminescent properties, is a good charge transporter, and efficiently injects the charge carrier of the electrodes [8,9]. Adam et al. [10,11] showed that some discotic liquid crystals, which are able to form a columnar phase, display an unusually high charge carrier mobility of the order of 10^{-2} cm^2 V^{-1} s^{-1}. However, in the initial applications of these liquid crystals in OLEDs the incorporation of the additional organic layer of a dye with electroluminescent properties was required [7,12]. Recently, Benning et al. [9,13] found that derivatives of 3,4,9,10-perylenetetracarboxylic acid exhibited not only fluorescent properties, but also may form a columnar hexagonal liquid crystalline phase over a certain temperature range; thus, they can be successfully used as organic layer in OLEDs [9].

In a previous paper we demonstrated that one of the compounds studied by Benning et al. [9,13],

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namely 3,4,9,10-tetra-(heptyloxy-carbonyl)-perylene, is able to form a LB film with efficient fluorescent properties. Somewhat later Antunes et al. [15] reported about the possibility of obtaining LB films from other perylene tetracarboxylic derivatives.

The first required step for successful LB film production is the creation of a stable, compressible monolayer at the air–water interface (Langmuir film) [4–6,16]. Previously, it was found that perylene and the majority of perylene derivatives can form Langmuir (L) films which can be easily transferred to the solid substrate (LB films), but only when they are mixed, usually with fatty acids [17–23]. Our studies [14], however, and those described by Antunes et al. [15] deal with the formation of L and LB films from pure perylene-like compounds.

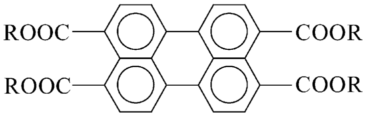
In this paper we have determined the L film formation ability for some derivatives of 3,4,9,10-tetracarboxylic acid and 3,4,9-perylenetricarboxylic acid. We also attempted to make L films from two other groups of organic compounds, namely naphthalene derivatives of bicarboxylic acid and derivatives of naphthoylenobenzimidazole. The compounds from these latter groups are dyes with good dichroic and excellent fluorescent properties [24–29] and can be used as guest species in passive and active guest-host liquid crystal displays (LCDs). If they are able to form stable and compressible L films, there exists the possibility of using them also as the luminescent layer in OLEDs construction.

2. Materials and methods

The following dyes were studied:

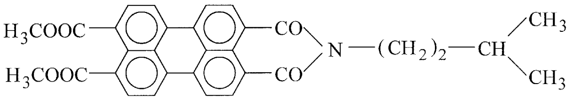
1. Derivatives of 3,4,9,10-perylenetetracarbolixylic acid

(a)

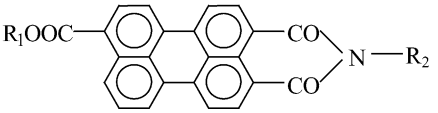


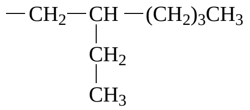
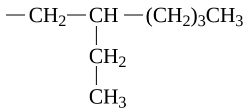
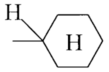
Dye code	R
1	$-(CH_2)_3CH_3$
2	$-(CH_2)_4CH_3$
3	$-(CH_2)_5CH_3$
4	$-(CH_2)_6CH_3$
5	$-(CH_2)_7CH_3$

(b) dye **6**



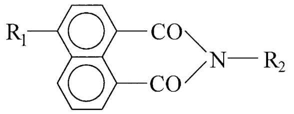
2. Derivatives of 3,4,9-perylenetricarboxylic acid



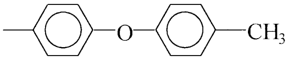
Dye code	R_1	R_2
7	 $-CH_2-\underset{\substack{\vert \\ CH_2 \\ \vert \\ CH_3}}{CH}-(CH_2)_3CH_3$	$-CH_2CH_3$
8	 $-CH_2-\underset{\substack{\vert \\ CH_2 \\ \vert \\ CH_3}}{CH}-(CH_2)_3CH_3$	$-(CH_2)_2OH$
9	$-(CH_2)_7CH_3$	$-(CH_2)_2CH(CH_3)_2$
10		$-(CH_2)_2CH(CH_3)_2$

3. Derivatives of naphthalenebicarboxylic acid

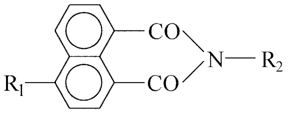
(a)

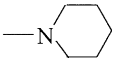
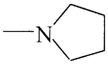
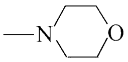
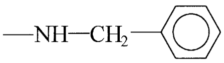
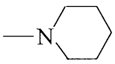
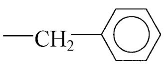
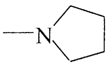
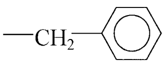
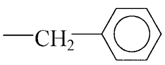
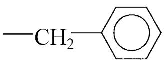
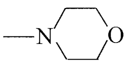
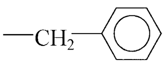
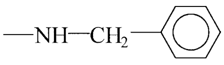
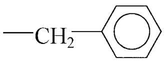


Dye code	R_1	R_2
11	$-NHCH_3$	$-CH_2CH_3$
12	$-NHCH_2CH_3$	$-CH_2CH_3$
13	$-NH(CH_2)_2CH_3$	$-CH_2CH_3$
14	$-NH(CH_2)_3CH_3$	$-CH_2CH_3$
15	$-NH(CH_2)_4CH_3$	$-CH_2CH_3$
16	$-NH(CH_2)_6CH_3$	$-CH_2CH_3$
17	$-NH(CH_2)_{11}CH_3$	$-CH_2CH_3$
18	$-NH(CH_2)_2CH(CH_3)_2$	$-CH_2CH_3$

19	$-N(CH_3)_2$	$-CH_2CH_3$
20	$-N(CH_3)_2$	$-(CH_2)_3CH_3$
21	$-N(C_8H_{17})_2$	

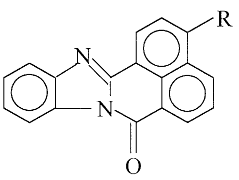
(b)



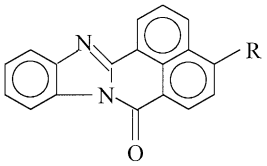
Dye code	R_1	R_2
22		$-(CH_2)_3CH_3$
23		$-(CH_2)_3CH_3$
24	$-NH(CH_2)_3CH_3$	$-(CH_2)_3CH_3$
25	$-N(CH_3)_2$	$-(CH_2)_3CH_3$
26		$-(CH_2)_3CH_3$
27		$-(CH_2)_3CH_3$
28		
29		
30	$-NH(CH_2)_3CH_3$	
31	$-N(CH_3)_2$	
32		
33		

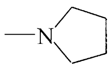
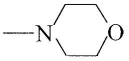
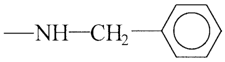
4. Derivatives of naphthoylenebenzimidazole

(a)



(b)



Dye code		R
(a)	(b)	
34	39	$-NH(CH_3)_2$
35	40	
36	41	
37	42	
38	43	$-NH(CH_2)_2CH_3$

All the dyes were synthesized and chromatographically purified at the Institute of Dyes, Łódź University of Technology, Poland.

In order to obtain Langmuir films the dyes investigated were dissolved in chloroform (Uvasol, spectroscopic grade, Merck) at a concentration of 0.1 mM in order to obtain stock solutions. The concentration of the solutions was confirmed spectroscopically. The solutions were prepared at room temperature shortly before spreading at the air–water interface.

Monolayer studies were performed using a Minitrough 2 (KSV Instruments) equipped with two barriers for monolayer compression, a Wilhelmy plate balance for surface pressure determination and a temperature control system. The subphase was deionized water obtained from a Mili-Q, Millipore water purification system. A constant subphase temperature was maintained by a cooling circulator and kept constant at 22 °C. The surface of the water in the trough of area 22 950 mm^2 (306×75 mm^2) was carefully purified using an aspirator and then the appropriate amount (from 70 to 150 μl) of the mixed solution was spread drop-wise from a microlitre syringe (Hamilton, England). The chloroform was allowed to evaporate for about 15 min after spreading and then the floating layer was slowly compressed, symmetrically from both sides at a barrier motion speed of 5 mm min^{-1} (approximately 2×10^{-7} nm^2 molecule^{-1} s^{-1}) while the surface pressure (π) versus area per molecule (A) isotherm was recorded. Stability tests were carried out for the L films checking the area per molecule variation to keep the pressure constant over 1 h. Ensuing L films were found to be stable and the data for π–A isotherms were acquired on a PC and processed using software provided by KSV. The isotherms were reproducible within an error of 0.02 nm^2 molecule^{-1}. Each isotherm was obtained by averaging five runs.

3. Results

The surface pressure–mean molecular area (π–A) isotherm measurement is a basic and widely used technique to characterize a Langmuir film. The π–A isotherm is a plot of the change in surface pressure (a measure of the change in surface tension of the water covered with monolayer with respect to pure water) as a function of the average area available for each molecule on the water surface. For the compounds investigated the isotherms were recorded both during compression and expansion processes and no significant differences in the isotherm runs were found. This means that equilibrium conditions were obtained. However, it was found that some of the dyes produced monolayers which were unable to offer any significant resistance to barrier compression: the molecules were expelled and the bulk phase was observed to form.

Figs. 1–3 show the characteristic π–A isotherms for some selected dyes, which were able to form a compressible and stable monolayer. The increase in π begins at the area equal to A_0, which is the first edge of the phase transition—at this moment

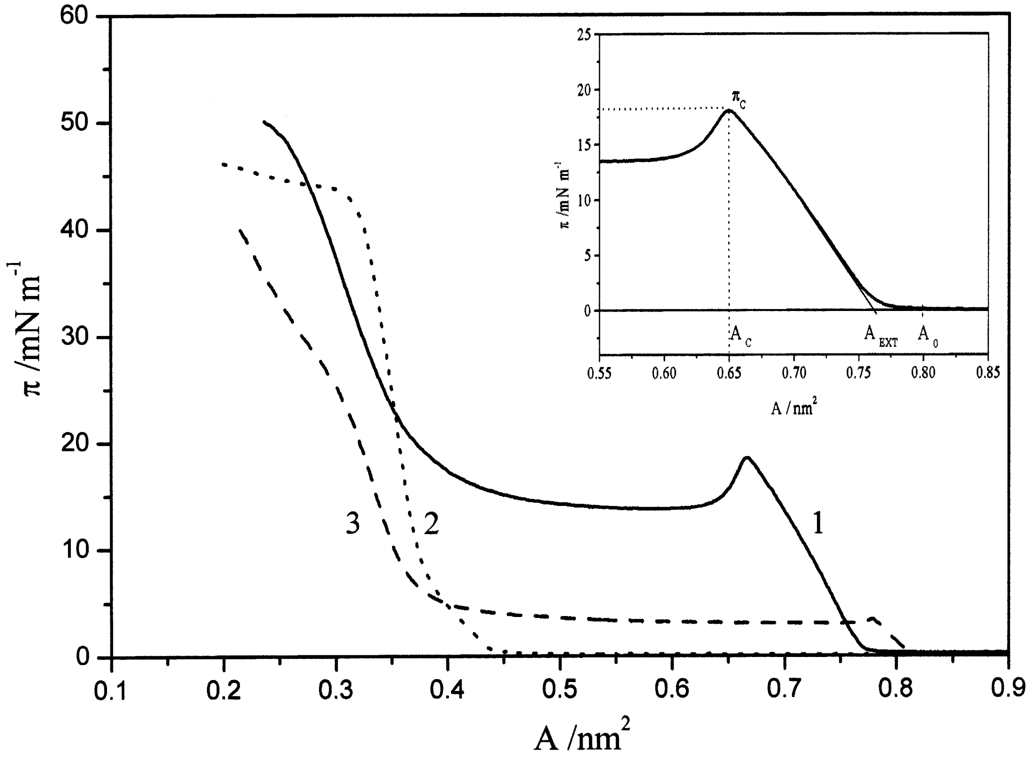


Fig. 1. Surface pressure–area isotherms of Langmuir films of some perylene derivatives: **2** (1), **7** (2) and **9** (3). In insert the values given in Tables 1–3 are determined.

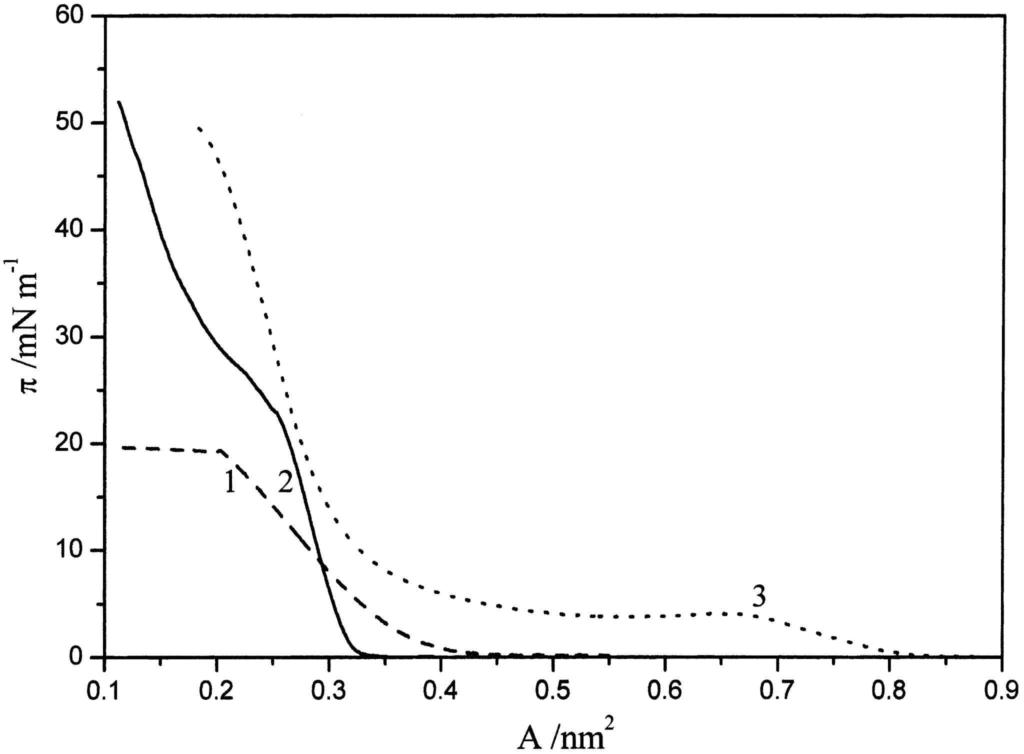


Fig. 2. Surface pressure–area isotherms of Langmuir films of some naphthalene derivatives: **15** (1), **17** (2) and **21** (3).

the monolayer begins to form. Tables 1–3 contain the values of A_0 and A_{EXT} (obtained by extrapolating the tangent of the tilt angle of π–A curve to $\pi = 0$—see insert in Fig. 2) for all the compounds investigated. In these tables the values of the collapse area (A_{C}) and the collapse pressure (π_{C}) are also given. The collapse point is recognized as the point in the isotherm where the ratio $\partial\pi/\partial$A begins to decrease due to the next phase transition. The nature of the isotherms are different for various dyes. In some cases, behind the collapse point a broad plateau region is observed, while in other cases, the pressure rises monotonically with a decrease in the available area. However, the shape of all π–A isotherms obtained for the dyes investigated can be found in literature for other compounds [15,16,30–34].

Table 1
Values of the area per molecule, collapse pressure and the angle between the normal to the water surface and rigid molecular cores of perylene derivatives

Dye code	A_0/nm^2	A_{EXT}/nm^2	A_{C}/nm^2	π_{C}/mN m^{-1}	δ/°
1	0.68	0.66	0.62	6.1	32
2	0.80	0.77	0.65	18.3	38
3	0.83	0.80	0.71	13.2	40
4	0.87	0.85	0.76	13.1	42
5	1.02	0.98	0.89	12.3	50
6	0.48	0.45	0.32	41.9	20
7	0.48	0.44	0.30	44.0	20
8	0.72	0.71	0.65	14.1	33
9	0.82	0.81, 0.41	0.78, 0.32	3.5, 25.0	38, 22
10	0.49	0.44	0.30	41.4	20

4. Discussion

4.1. Derivatives of 3,4,9,10-perylenetetracarboxylic acid and 3,4,9-perylenetricarboxylic acid

From the results presented in Table 1 it is evident that all the perylene derivatives investigated

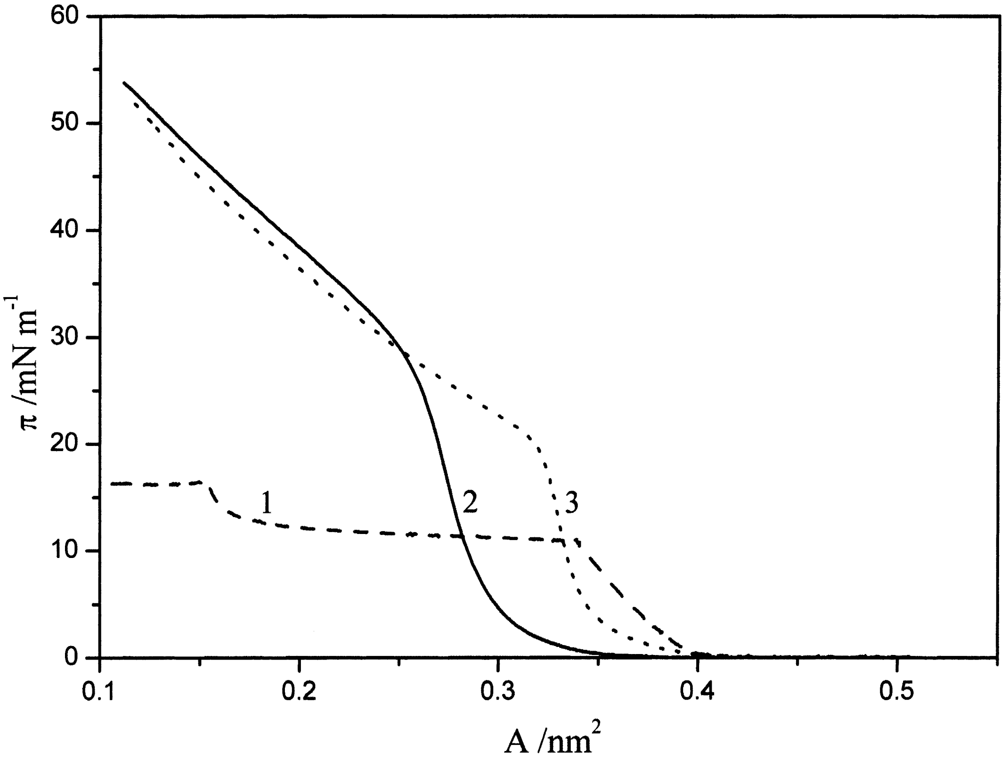


Fig. 3. Surface pressure–area isotherms of Langmuir films of some naphthoylenebenzimidazole derivatives: **35** (1), **42** (2) and **43** (3).

in this work were able to form a stable and compressible monolayer at the air–water interface. A cross-sectional area of the perylene skeleton with four –C=O groups calculated on the basis of the space-filling model, including van der Waals radii of the terminal atoms [35], was about 1.30 nm^2, while cross-sectional areas estimated from the longer and shorter edge-on configuration (assuming free rotation) were, respectively, 1.58 and 0.66 nm^2. Therefore, it is clear from the results listed in Table 1 that the molecules of the perylene derivatives neither assume an edge-on configuration with the possibility of free rotation, nor lie flat at the water surface. The values of the areas found from the isotherms suggest that in the monolayer, the rigid cores of the molecules must be tilted with respect to the water surface. Table 1 contains the values of the tilt angle, δ, which was estimated as the angle between the rigid molecular core of **1–10** and the normal to the water surface. It is clear that the alignment of the molecules in the L films is strongly affected by the molecular structure of the compound investigated. Among the symmetrically substituted dyes, **1–5**, the angle δ increased with increase in the length of the alkoxy chains. However, with the exception of dye **1**, an increase in chain length lowered the collapse pressure value and increased the isotherm slope, which indicates that both the stability and the rigidity of the monolayer decreased.

Compression of the L film of dyes **2–5** beyond the collapse point resulted in a broad plateau region (curve 1 in Fig. 1), the breadth of which rose with n. In this region, π remained constant with decreasing A. When A reached a value of about 0.35–0.45 nm^2 (depending on n), a rapid rise in π occured. Dye **1**, with the shortest alkoxy chain, behaved dissimilarly with respect to compounds **2–5**. The π_C value was very small and the second rise in surface pressure occurred at significantly larger area ($\approx$0.55 nm^2—data not shown) than in the case of other dyes. This indicates that the monolayer formed by **1** was very fragile and unstable.

Table 2
Values of the area per molecule, collapse pressure and the angle between the normal to the water surface and rigid molecular cores of naphthalene derivatives

Dye code	A_0/nm^2	A_{EXT}/nm^2	A_C/nm^2	π_C/mN m^{-1}	δ/°
11	–	–	–	≈0	–
12	–	–	–	≈0	–
13	–	–	–	≈0	–
14	–	–	–	≈0	–
15	0.44	0.37	0.20	19.3	27
16	0.44	0.40	0.30	19.5	30
17	0.34	0.31	0.26	22.0	23
18	0.46	0.36	0.20	20.6	26
19	–	–	–	≈0	–
20	–	–	–	≈0	–
21	0.84	0.81	0.70	3.7	90
22	–	–	–	≈0	–
23	–	–	–	≈0	–
24	0.45	0.40	0.32	16.2	30
25	–	–	–	≈0	–
26	–	–	–	≈0	–
27	0.36	0.33	0.31	5.3	24
28	0.47	0.46	0.43	2.0	35
29	0.35	0.25	0.20	24.4	18
30	0.48	0.40	0.35	12.0	30
31	–	–	–	≈0	–
32	–	–	–	≈0	–
33	0.46	0.41	0.36	9.2	30

Previously, it was found that some discotic liquid crystals can form an edge-on arrangement at the air–water interface leading to the building of the columns due to the strong attractive interaction between the rigid cores [30–31]. As compounds **1–5** are able to form a discotic (columnar) phase over an elevated temperature region [13], it is reasonable to assume that they also have the tendency to create columns on the water surface during compression. When A decreases, some of columns 'jump out' above the monolayer surface and form the next layer. The second rise in π could then be explained as the formation of a regular multilayer. Another possibility is that in the plateau region, the dye molecules which create a tilt δ at the beginning of the monolayer formation, assume a more vertical arrangement as the available area per molecule decreases. A second rapid rise in π would then indicate the creation of a rigid condensed layer.

Table 3
Values of the area per molecule, collapse pressure and the angle between the normal to the water surface and rigid molecular cores of naphthoylenebenzimidazole derivatives

Dye code	A_0/nm^2	A_{EXT}/nm^2	A_C/nm^2	π_C/mN m^{-1}	δ/°
34	37.6	0.36	0.30	10.7	26
35	41.5	0.39	0.34	11.2	29
36	–	–	–	≈0	–
37	37.5	0.31	0.20	23.0	23
38	45.1	0.37	0.34	15.8	27
39	37.0	0.33	0.27	6.1	24
40	40.7	0.37	0.32	12.6	27
41	–	–	–	–	–
42	36.2	0.30	0.27	27.0	22
43	42.0	0.35	0.33	20.4	26

For dye **6** and the unsymmetrically substituted perylene derivatives (**7–10**), the appearance of a liquid crystalline phase was not found, but the results presented in Table 1 indicate that, as with dyes **1–5**, molecules of **6–10** form a tilted, edge-on arrangement without the possibility of free rotation at the air–water interface. The L films of dyes **6**, **7** and **10** were characterized by very high surface pressure π_C suggesting the formation of the stable and rigid monolayer. These three dyes displayed a strong tendency to align their cores vertically to the water surface ($\delta = 20°$). The molecular structure of dye **8** differs from that of dye **7** only in terms of the R_2 substituent. The presence of the –OH group in the chain of dye **8** lowered the π_C value and, simultaneously, the slope of the molecular core with respect to the water surface. Dye **9** showed a non-typical form of π–A isotherm of the L film with respect to that of dyes **6**, **7** and **10** (compare curves 2 and 3 in Fig. 1). After a small increase in π up to the collapse point at $A \approx 0.8$ nm^2, a broad plateau region was observed, and again at $A \approx 0.4$ nm^2, a second rapid rise of π occured. Therefore, in Table 2, two values of A_{EXT}, A_C and π_C are given for this dye which indicate that already at significantly large area, the molecules of dye **9** are able to be compressed, but molecular packing is rather small. However, when the available area is reduced a rigid condensed layer was formed, in which the molecules were densely packed in tilted 'stacks', as was found for dyes **6**, **7** and **10**.

4.2. Derivatives of naphthalenebicarboxylic acid

The cross-sectional area of the naphthalene skeleton with two –C=O groups, calculated in the same manner as in the case of the perylene derivatives, was 0.84 nm^2, whereas the cross-sectional area estimated from an edge-on configuration, by assuming the free rotation of the molecule, was 0.66 nm^2. Therefore, the results of A_{EXT} presented in Table 2 show that the rigid cores of the naphthalene derivatives must be tilted with respect to the water surface; the tilt angles δ are given in Table 2.

Not all derivatives of naphthalenebicarboxylic acid are able to form a compressible monolayer at the air–water interface. This ability is strongly dependent on the molecular structure of two side substituents. Although the relation between substituent molecular structure and the ability to form a L film is not simple, some indications can be made. The compressible monolayer at the water surface cannot be formed before the length of the alkyl chain achieves a suitable value. This is evident for dyes **11–17**, which differ only in the length of R_1. Contrary to the perylene derivatives, the value of π_C and the slope of the isotherm increased here with increase in chain length (curves 1 and 2 in Fig. 2) providing evidence about the increase in the monolayer stability and rigidity. The molecules of dye **18** have a different R_1 substituent, and although it is relatively short, a L film was created. However, the small slope of the π–A isotherm suggests that the monolayer was very fragile and unstable. Comparing the data for dye **14** with those for dye **24** it is clear that the length of the R_2 substituent affects the ability to form a compressible L film. Thus, not only is the length of the chain important, but the whole molecular structure of both substituents. When, R_1 or R_2 contain a benzene ring, the creation of a stable monolayer is more probable (compare the data for dyes **22**, **23** with those for dye **27** and dyes **28**, **29**). It should be noted that dyes with groups $-N(CH_3)_2$ or a 1-oxa-4-azocyclohexane ring in R_1 were not able to form a L film, independent of the structure of R_2. Dye **21**, which of all the dyes investigated had an A_{EXT} value comparable to the cross-sectional area of the naphthalene skeleton, behaves non-typically (curve 3 in Fig. 2). The value of A_{EXT} suggests that in the monolayer, the rigid cores of the molecules of the dye lie flat on the water surface. Such a molecular alignment can be related to the presence of two benzene rings in the R_2 substituent, which, in the first stage of the monolayer formation, do not allow the assumption of a tilted arrangement of the molecules.

4.3. Derivatives of naphthoylenebenzimidazole

As in the case of the naphthalene derivatives of bicarboxylic acid, the cross-sectional area of the rigid cores of the naphthoylenebenzimidazole derivatives can be assumed to be equal to 0.84 nm^2. Therefore, it is clear from Table 3 that, as in previous cases, for these dyes, a tilted arrangement of the molecules in the L film occurred. The calculated δ angles are given in Table 3. All the values of δ are similar to each other, although both the π_C values and the isotherm slopes (Fig. 3) are different for various dyes, indicating the influence of the substituent molecular structure on both monolayer stability and rigidity. When in R the 1-oxa-4-azocyclohexane ring (dyes **36** and **41**) occurred, the monolayer could not be formed, as was found for naphthalene derivatives. It should be noted that in the case of derivatives of naphthoylene-benzimidazole, an increase in the alkyl chain length as well as the presence of a benzene ring in the substituent, increased the Langmuir film stability (compare the results of π_C for dyes **34**, **39** with those of **38**, **43** and for dyes **37**, **42** with others). The properties of the monolayer do not depend significantly on the position of the R substitution.

5. Conclusions

It has been shown that it is possible to prepare stable Langmuir films from derivatives of: 3,4,9,10-perylenetetracarboxylic acid, 3,4,9-perylenetricarboxylic acid, naphthalenebicarboxylic acid and naphthoylenebenzimidazole. It has also been found that the ability to form compressible monolayer at the air–water interface depends on the molecular structure of the substituents which influences also both the stability and rigidity of the monolayer. The rigid cores of molecules of dyes investigated show a tendency to align at a tilted angle with respect to the water surface.

To the best of our knowledge these findings of derivatives of naphthalenebicarboxylic acid and naphthoylenebenzimidazole have not been previously reported in literature. The fact that dyes other than perylene derivatives with good fluorescent properties are able to form stable and compressible monolayer at the air–water interface, provides new perspectives for the application of such compounds not only as guest species in LCDs but also as active layers in OLEDs.

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