

Research Article

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**FORMATION OF CdSe/CdS/ZnS QUANTUM DOTS FLOATING MONOLAYERS
ON THE NiCl₂ CONTAINING SUBPHASE**

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Abstract. *The development of low-defect self-organizing thin film materials is one of the most important directions of modern optoelectronics. For this purpose, the formation and stability of quantum dots (QDs) floating monolayers on the composition (pure water or NiCl₂ aqueous solutions) and temperature (19 and 25 °C) of subphase have been investigated. The studied quantum dots were CdSe/CdS/ZnS (core/shell/shell) stabilized by oleic acid. The data on π -A isotherms, hysteresis, and stability of QDs floating monolayers were analyzed. It was found that the monolayer formed on the pure water subphase is more stable. It was established that the increase of NiCl₂ content in the aqueous subphase (from 1 to 5 mM) leads to the decrease of the gas phase region and the increase of the liquid and condensed phase regions on π -A isotherms. It was suggested that Ni²⁺ cation interacts with the carboxylic groups of free (unbounded to QDs) oleic acid molecules. As a result, the density of floating layer and the compression module values increased. The highest compression modulus values of QDs floating monolayers were found for the 5 mM NiCl₂ containing subphase. On the basis of the studied floating layers, thin films were obtained on silicon substrates, and their surface was investigated by the AFM method. It was determined that the presence of NiCl₂ in the aqueous subphase leads to an increase in the thicknesses and hydrophobicity of the prepared thin films. The obtained data can be used at the formation of multicomponent thin film nanomaterials containing quantum dots.*

Key words: quantum dots, floating layers, Langmuir-Schaefer method, thin film, AFM, oleic acid, NiCl₂, mechanical properties

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ФОРМИРОВАНИЕ ПЛАВАЮЩИХ МОНОСЛОЕВ КВАНТОВЫХ ТОЧЕК CdSe/CdS/ZnS НА ПОВЕРХНОСТИ СУБФАЗЫ, СОДЕРЖАЩЕЙ NiCl₂

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Аннотация. Создание малодефектных самоорганизующихся тонкопленочных материалов является одним из важнейших направлений развития современной оптоэлектроники. С этой целью были исследованы формирование и стабильность плавающих монослоев квантовых точек (КТ) в зависимости от состава (чистая вода или водные растворы NiCl₂) и температуры (19 и 25 °С) субфазы. Исследуемые квантовые точки CdSe/CdS/ZnS (ядро/оболочка/оболочка) были стабилизированы олеиновой кислотой. Проанализированы данные π -А изотерм, гистерезиса и стабильности плавающих монослоев. Было обнаружено, что плавающий монослой КТ, сформированный на субфазе чистой воды, является более стабильным. Установлено, что увеличение содержания NiCl₂ в водной субфазе (с 1 до 5 мМ) приводит к уменьшению области газовой фазы и увеличению областей жидкой и конденсированной фаз на π -А изотермах. Было высказано предположение, что катион Ni²⁺ взаимодействует с карбоксильными группами свободных (не связанных с КТ) молекул олеиновой кислоты. В результате увеличивается плотность плавающего слоя и значения модуля сжатия. Наибольшие значения модуля сжатия плавающих монослоев КТ были обнаружены для субфазы, содержащей 5 мМ NiCl₂. На основе исследованных плавающих слоев были получены тонкие пленки на кремниевых подложках; поверхность пленок исследована методом АСМ. Установлено, что присутствие NiCl₂ в водной субфазе приводит к увеличению толщины и гидрофобности тонких пленок. Полученные данные могут быть использованы для формирования многокомпонентных тонкопленочных наноматериалов, содержащих квантовые точки.

Ключевые слова: квантовые точки, плавающие слои, метод Ленгмюра-Шеффера, тонкие пленки, АСМ, олеиновая кислота, NiCl₂, механические свойства

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Introduction

Currently, Langmuir-Blodgett (LB) technique is used to create materials for optoelectronic devices such as solar cells [1–3]. The main idea of the technique is the self-organization of amphiphilic (surfactant) molecules at the water–air interface giving objects with a high degree of structural perfection. LB technique allows to control the required properties of floating layer

(packing density) and, therefore, to tune the properties of deposited thin film.

The presence or absence of ions in a subphase can also influence properties of forming floating layers. It was shown that when metal ions are added to the subphase during the formation and compression of a surfactant monolayer, the carboxyl group of surfactant molecule interacts with metal ion in the subphase and forms a salt [4]. In this case, the π -A isotherm shifts

to the right, regarding to the formation of a monolayer on the surface of pure water [5].

One of the promising materials for creating floating (Langmuir) monolayers are semiconductor quantum dots (QD) that have found their application in various fields of science and technology [6]. QDs possess unique optical and electrical characteristics that result in the manifestation of a quantum-size effect, i.e. dependence of particle properties on their size [7, 8].

The modification of a QDs monolayer can also be carried out by dissolving of various salts in the aqueous subphase [9, 10]. In this case, the floating monolayer of the QDs capped with fatty acid interacts with metal ion and salt of fatty acid is formed. As a consequence, the presence of metal ion in monolayer influences the properties of floating monolayer structure and then the resulting thin film transferred onto solid substrate.

By changing salt concentration and subphase temperature [5], it is possible to vary the surface morphology and composition of QDs floating monolayer as well as its mechanical (compressibility and compression modules) and electrical (surface potential) properties [5, 11, 12].

The characterization methods: measurement of π - A isotherm and atomic force microscopy provide the quantitative and qualitative information that could help to evaluate parameters of floating layers and thin films, correspondingly.

In this work, we studied the floating monolayers of CdSe/CdS/ZnS quantum dots formed at the air–water interface with varying content of NiCl₂ salt in an aqueous subphase. The presence of NiCl₂ in the subphase was aimed to get the highest packing density of QDs in floating monolayer. The effect of subphase temperature was used to study the stability of the floating monolayers.

Materials and methods

The studied QDs were synthesized by the group of Goryacheva I.Yu. according to the technique described in [13]. They represent 10⁻⁶ M colloidal chloroform solution of CdSe/CdS/ZnS QDs stabilized with oleic acid.

In the experiments we used deionized water (DW) with approximate resistance $R = 18 \text{ MOhm} \times \text{cm}$ as a subphase basis. The surface of a subphase was cleaned by suction to ensure that there were no contaminants. The subphase temperature was 19 or 25.0 with accuracy $\pm 0.5 \text{ }^\circ\text{C}$.

In order to add metal ions to the subphase, we prepared NiCl₂ (*Sigma Aldrich*) solutions in deionized water (DW) with concentrations varying from 10⁻³ to 5 × 10⁻³ M.

QDs floating monolayers were formed with the use of KSV Nima LB trough KN 2002 (*KSV Nima, Finland*). 50 μL of QDs solution were spreaded by a syringe over the subphase surface. Then we waited for 8 min until complete chloroform evaporation. The error of surface pressure did not exceed 0.03 mN/m. The monolayer was formed via LB technique using the compression barriers moving at a speed of 12 mm/min. The surface pressure isotherms were measured by two movable barriers located on top of the water surface (computer controlled LB trough).

For deposition experiments, the floating layers were exposed to surface pressures corresponding to gas, liquid expanded and condensed phases (0.3, 5.0 and 29 mN/m, respectively) [14]. Thin films were deposited onto solid substrates (silicon wafers, SiO₂-coated) at a rate of 2 mm/min by Langmuir-Schaefer method.

Atomic force microscopy (AFM) images were taken in a semi-contact mode using Solver Nano microscope (NT-MDT, Russia).

Results and discussion

π - A isotherms

With the aim to study the hysteresis behavior of QDs floating layers, two cycles of the compression and expansion isotherms were registered. The cycles accompanied by only slight changes in surface pressure indicating a good stability of the formed floating layers.

The hysteresis curves of π - A isotherms were obtained (Fig. 1). They show that the occupied area of QDs monolayer decreased during the compression and decompression cycles due to the shift of isotherm compression curve of the second cycle. The lift-off point (LP) of isotherm (it is the point when the isotherm curve starts to rise [15], Fig. 1) is shifted to the left. Correspondingly, the area of floating layer in this point decreases from 96.5 cm² to 83.1 cm². The occupied area in the condensed phase was extrapolated from the isotherm (Fig. 1) and calculated to be $54 \pm 0.1 \text{ cm}^2$.

With the aim to evaluate the effects of NiCl₂ in aqueous subphase on the QDs floating layer formation, the π - A isotherms were registered (Fig. 2). Concentration of NiCl₂ in the subphase was 1.0, 1.5, 2.0 and 5.0 mM. The data were compared with the case of pure water subphase.

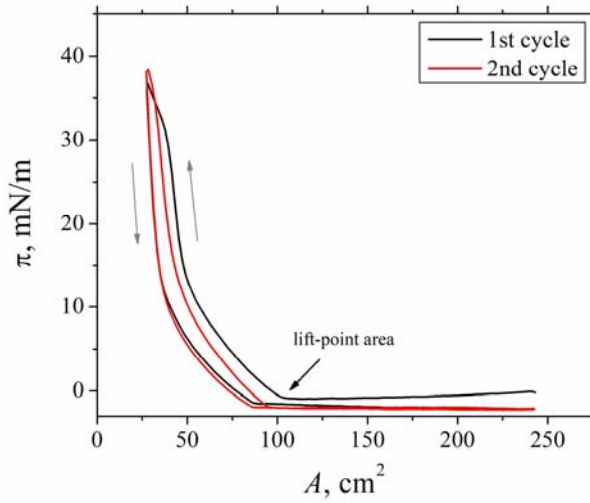


Fig. 1. Compression and expansion isotherms of the QDs floating monolayer on the pure water subphase

The following typical features of the π - A isotherms should be mentioned. At low surface pressure ($\pi \leq 1$ mN/m), QDs monolayers formed a gaseous-liquid coexistence phase before the lift-off area of monolayer (Fig. 2). In this case, QDs are mostly free in space. Then, the monolayers were forcibly compressed into a liquid phase (I). Further barrier compression caused a sudden phase transition to a condensed phase (II), when the hydrophobic part of free (unbounded to QDs) oleic acid molecules aligned almost perpendicular to the surface of subphase.

As can be seen from Fig. 2, the isotherms of QDs monolayers depend on the concentration of NiCl₂ in the subphase. The increase of NiCl₂ concentration leads to the decrease of gas phase region and the increase of liquid and condensed phase regions. For example, when $A = 27$ cm² the addition of NiCl₂ into subphase changes the surface pressure (π) by 28 %: from 35 to 50 mN/m. Probably, Ni²⁺ cation interacts with carboxylic groups of free oleic acid molecules (the oleic acid molecules, which are unbounded to QDs), that is the acid exchanges its hydrogen atom with nickel ion forming oleate salt: Ni(C₁₇H₃₃COO)₂ [2, 16]. Due to this fact, the density of floating layer and the compression module values are increased.

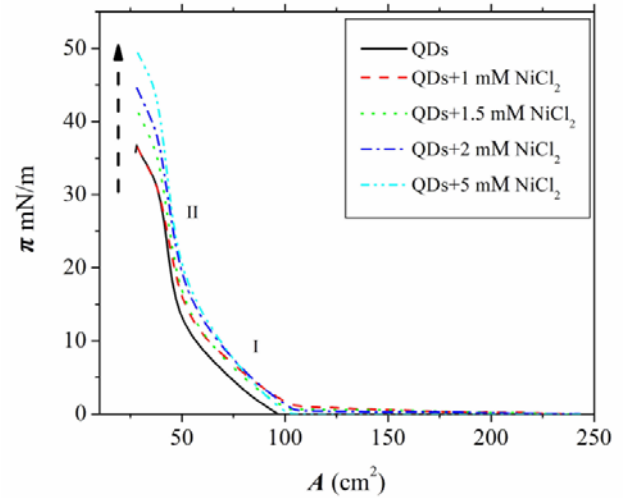


Fig. 2. π - A isotherms of QDs floating monolayers at different concentration of NiCl₂ in aqueous subphase. I – liquid phase, II – condensed phase. The dashed arrow shows the surface pressure increase on the addition of NiCl₂ into aqueous subphase

Mechanical properties of floating layers

The phase transitions in floating monolayer can be explained by studying the mechanical properties – compressibility (K) and compression modulus (χ):

$$\chi = \frac{1}{K}, K = -\frac{1}{A_0} \left(\frac{dA}{d\pi} \right), \quad (1)$$

where dA is the change in the area of Langmuir monolayer, $d\pi$ is the change in surface pressure value, A_0 is the monolayer area (condensed phase) [9, 17].

In the described series of experiments, the influence of NiCl₂ on the compression modulus of QDs monolayers was investigated (Fig. 3). The highest values of the compression modulus are equaled to 359.4 and 42.4 mN/m for the subphase containing 5 mM NiCl₂ for regions I and II, respectively (Fig. 2). The lowest values of the compression modulus were 86 and 35 mN/m for regions I and II, respectively – for the pure water subphase. According to literature data, the values of the compression modulus of floating monolayer of 12.5–50 mN/m correspond to liquid-expanded phase [18, 19], the range of 100–250 mN/m is typical for liquid-condensed phase, and the values above 250 mN/m usually observed in condensed phase [20].

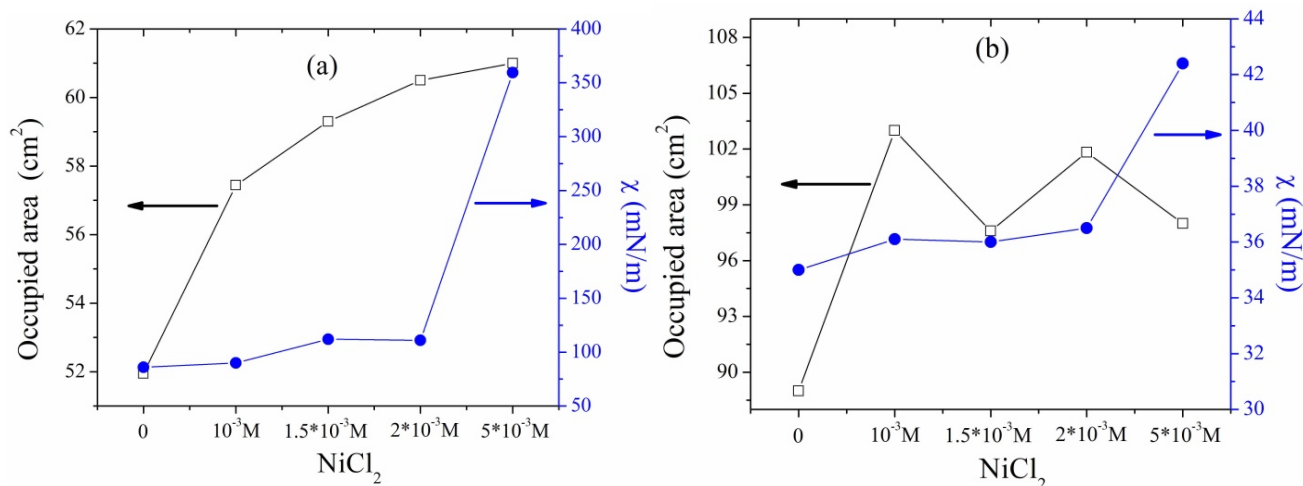


Fig. 3. The dependences of occupied area (—□—) and compression modulus (—●—) of QDs floating monolayers on the concentration of NiCl₂ in the aqueous subphase. a) condensed phase (region II, Fig. 2) and b) liquid phase (region I, Fig. 2)

Floating monolayer stability

To study the monolayer stability, the QDs floating layers were compressed at the surface pressure value of 20 mN/m with the maximum forward/backward movement rate of barriers of 1 mm/min, and the decrease of the monolayer area over time (1800 s) was evaluated (Fig. 4, a). The experimental results of relaxation were approximated by a double exponent of the relaxation components (τ_1 and τ_2). These individual parameters can be extracted from the exponential dependences as two terms of the equation:

$$A/A_0(t) = a_1 \exp(-t/\tau_1) + a_2 \exp(-t/\tau_2), \quad (2)$$

where A/A_0 is the relaxation area, a_1 and a_2 are dimensionless constants, t is the variable time (0–1800 s), τ_1 and τ_2 are the equilibrium times of the regions I and II, correspondingly (Fig. 2).

Fig. 4, a shows the QDs floating monolayer stabilities at different conditions (subphase content and subphase temperature). At 25 °C, the decrease of monolayer area by the end of observation time was about 17% for both concentrations of NiCl₂ (1 and 5 mM). It is somewhat different from the case with the pure water subphase (13.5%). Therefore, the monolayer formed on the surface of pure water subphase is the most stable.

At 19 °C, the floating monolayer area decreased by 12.8 and 12.9 % for pure water and 1 mM NiCl₂ subphase, respectively. Thus, the decrease of subphase temperature improved the stability of monolayers formed on the NiCl₂-containing subphase. It became similar to the values obtained for pure water subphase.

The data presented in Table 1 evidence that the presence of NiCl₂ in the subphase causes a significant decrease in the equilibrium times in comparison with the pure water subphase at both temperatures (19 and 25 °C). The increase in the concentration of NiCl₂ from 1 to 5 mM has only negligible influence. Parameter τ_1 occurred to be more sensitive to the temperature of the subphase, than τ_2 . Such behavior indirectly tells about two relaxation processes having different nature.

Surface potentials of floating monolayers on different subphases at different temperatures changing over time at a fixed surface pressure of 20 mN/m are presented in Fig. 4, b. It can be seen that the lowest value of the surface potential is observed at 25 °C on pure water surface, it decreases by 11.4 % over time. The highest values of the surface potential were found for the floating monolayer formed on the subphase with 1 mM of NiCl₂ at 19 °C starting from ~500th up to 1800th seconds of observation. The values for other floating monolayers lay between the two above mentioned data.

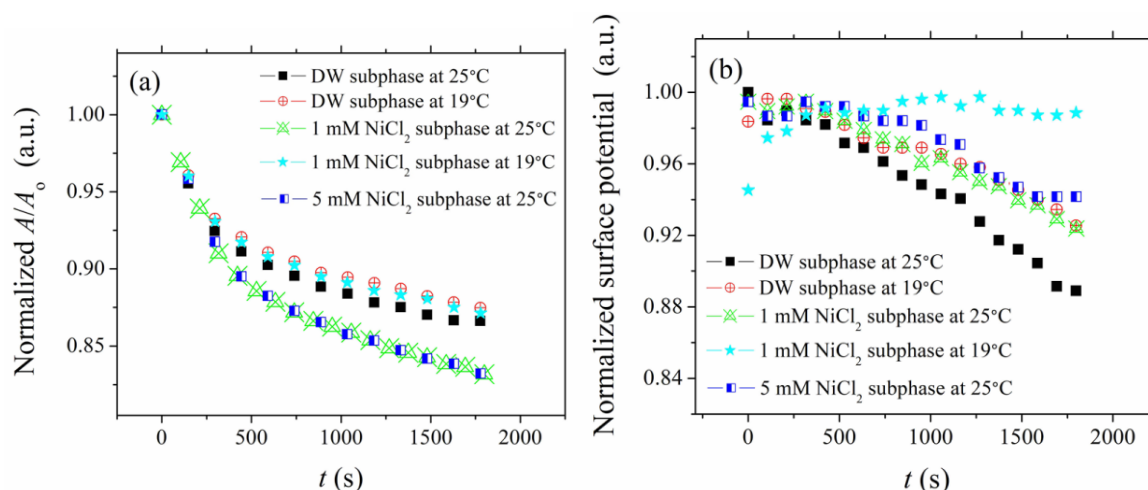


Fig. 4. Floating monolayer stabilities (a) and surface potentials (b) at different NiCl₂ concentrations in the aqueous subphase

Table 1. Equilibrium times of QDs monolayer area at different conditions

Subphase	T, °C	τ_1 , s	τ_2 , s
DW	25	760	580
DW	19	1770	640
1 mM NiCl ₂	25	590	470
1 mM NiCl ₂	19	1640	630
5 mM NiCl ₂	25	610	490

Thin films on solid substrates

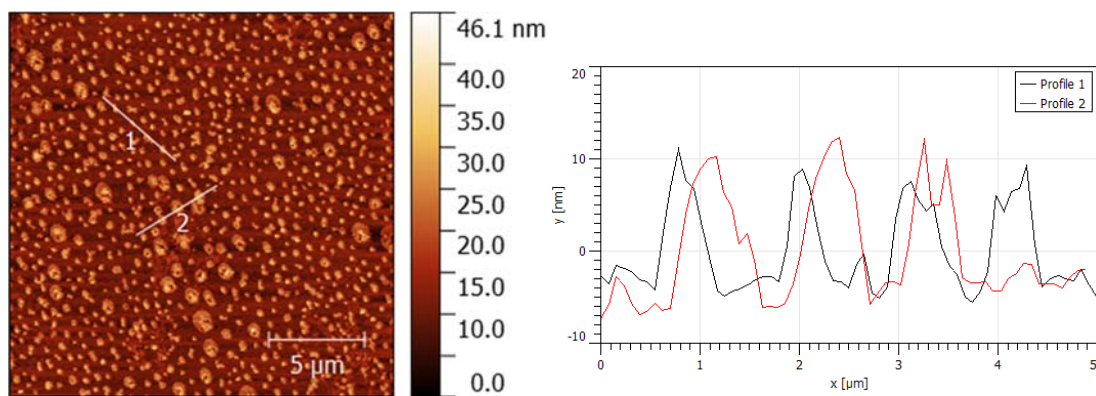
As a rule, a floating monolayer transferred onto a solid substrate by the Langmuir-Schaefer method (it is when the substrate is oriented horizontally) retains the structure that it had during transfer. This feature (assumption) was used to perform a more detailed study of the dissolved salt effect on the morphology of a QD thin film on a solid substrate.

Floating QD monolayers were transferred from pure water surface and surface of the 1 and 5 mM of NiCl₂ solutions onto monocrystalline silicon plates by Langmuir-Schaefer technique. The surface pressure during transfer was from 0.3 to 5 mN/m. Then, the obtained films were studied by atomic force microscopy (AFM). Relief and individual relief profiles are presented in Fig. 5.

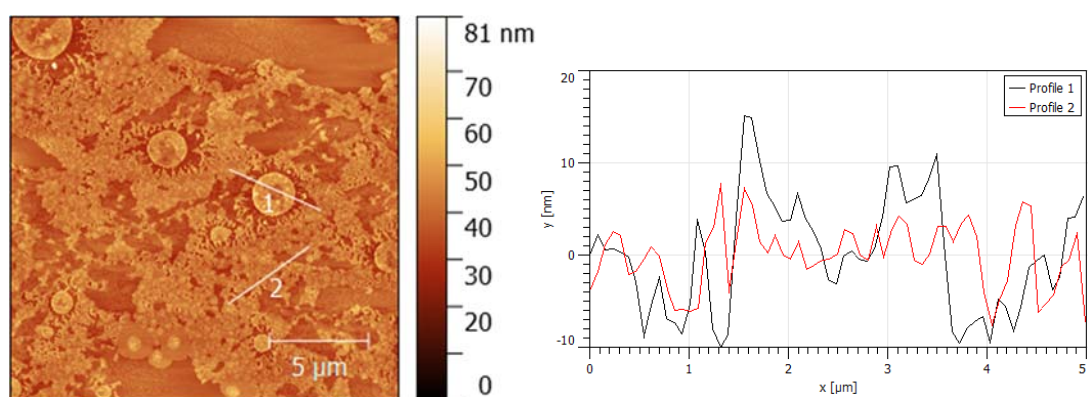
The AFM images show various types of layer formation throughout the QDs monolayer compression. With an increase in the value of the surface pressure from 0.3 to 5 mN/m, the total surface area decreases. This is caused by the decrease in the distance between

quantum dots in the film (up to formation of aggregates due to the poor QDs – water interaction) and the shrinking of the “empty” regions (the regions, which are free from the QDs but containing oleic acid molecules unbounded with QDs). The presence of empty areas in the films obtained when the surface pressure was 5 mN/m indicates that the colloid solution of QDs contained an excessive amount of oleic acid. The root mean square (RMS) roughness value of the monolayer film transferred from the pure water subphase increased from 5.3 to 8.3 nm. While, in the case of the 5 mM NiCl₂ containing subphase, the RMS roughness increased from 5.6 to 8.4 nm (see the corresponding profiles in Fig. 5 and Fig. 6). With further compression and reaching the surface pressure of 30 mN/m, the floating layer enters the pre-collapse "transitional" state.

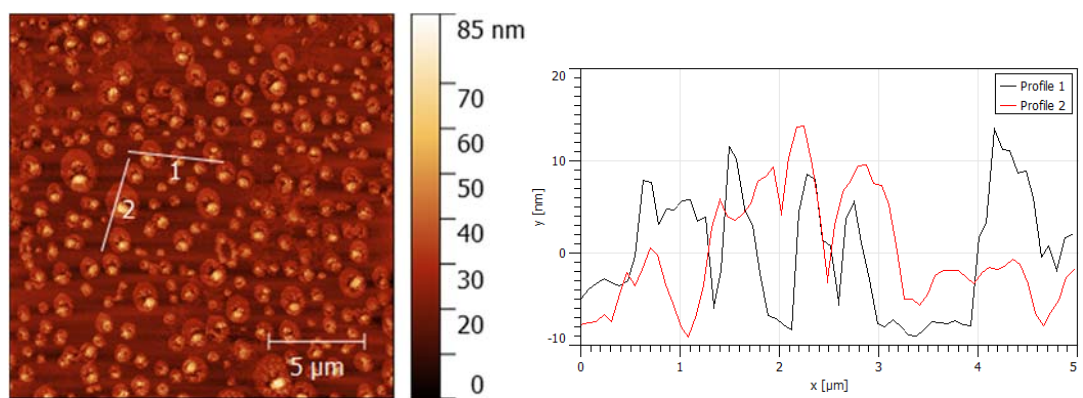
The AFM data of films obtained at two surface pressures (0.3 and 5.0 mN/m) support our suggestions about the presence of two states of floating monolayers (I and II phases, Fig. 2), exhibited regardless of the presence of salt in the subphase.



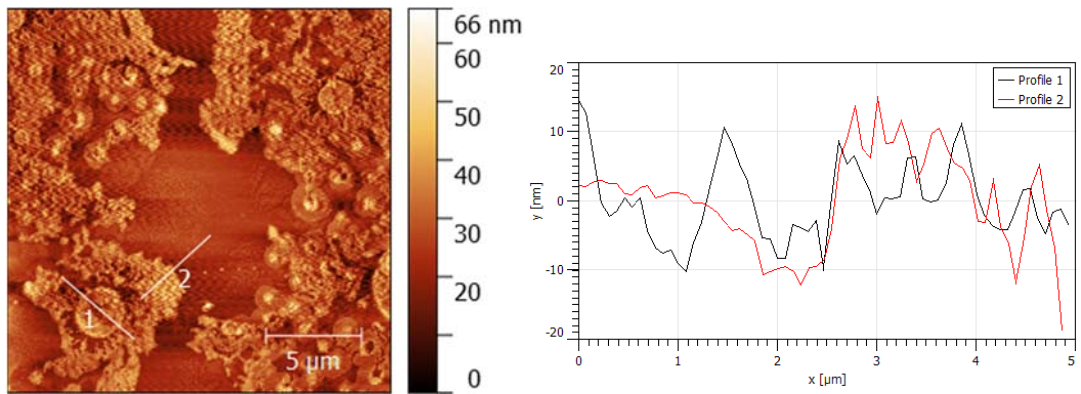
a – 0.3 mN/m, DW subphase



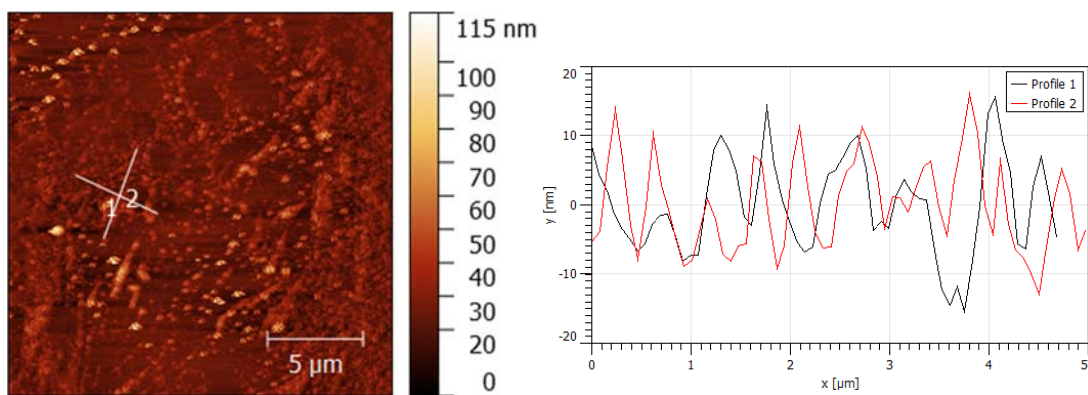
b – 5.0 mN/m, DW subphase



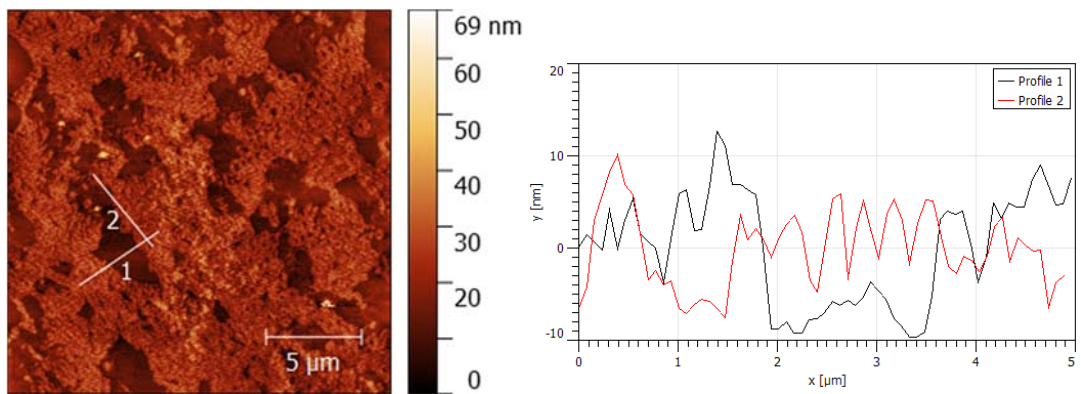
c – 0.3 mN/m, 1 mM NiCl₂ subphase



d – 5.0 mN/m, 1 mM NiCl₂ subphase



e – 0.3 mN/m, 5 mM NiCl₂ subphase



f – 5.0 mN/m, 5 mM NiCl₂ subphase

Fig. 5. AFM images and profiles of QDs thin films at different surface pressures and NiCl₂ concentrations

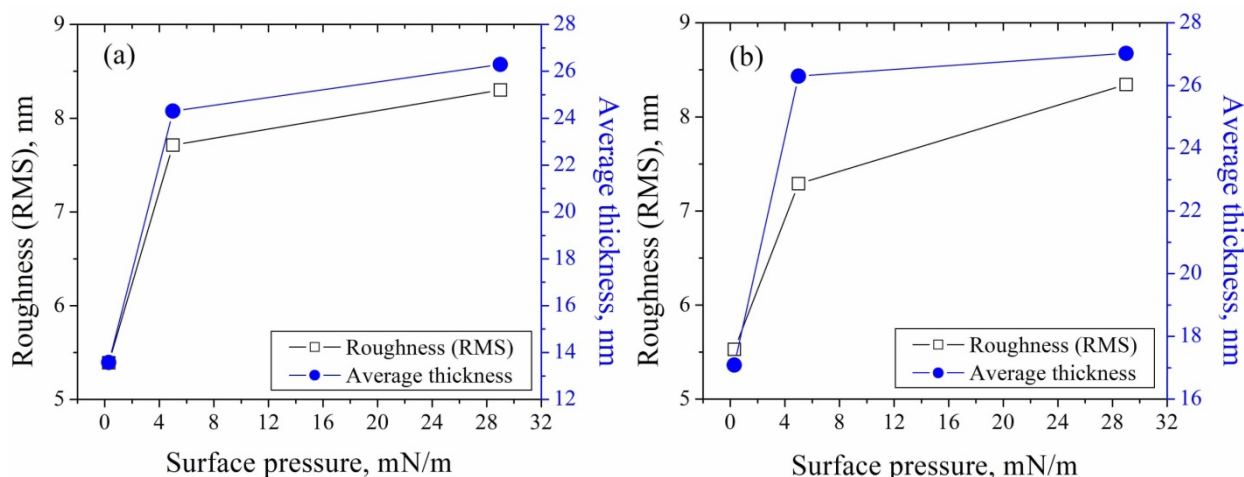
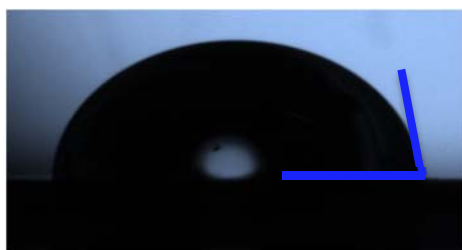


Fig. 6. RMS roughness value and average thickness of QDs thin films deposited on silicon substrates at different surface pressures spreaded of the water subphase (a) and NiCl₂ containing subphase (b)

In order to analyze the effect of subphase composition on the hydrophobicity of the QDs thin films transferred onto silicon substrate, the wetting angles of the obtained films were evaluated by the lying drop method. As it can be seen from Fig. 7, the average contact angle changes from 88.2° to 92.8° (by more than

5 degrees) for the films deposited on the silicon substrate from the pure water and the 5 mM NiCl₂ containing aqueous subphase, correspondingly. An increase in the contact angle indicates a decrease in the surface energy of thin film.



a



b

Fig. 7. The contact angles of QDs thin films deposited on silicon substrate from: a – the pure water subphase, b – the NiCl₂ containing subphase

Conclusions

The effect of the composition (pure water or NiCl₂-containing aqueous solutions) and temperature (19 and 25 °C) of subphase on the formation of floating monolayers by CdSe/CdS/ZnS quantum dots and the structure of their thin films transferred onto solid substrate has been studied.

Hysteresis behavior was studied by the compression and expansion isotherm method on the pure water subphase. It was found that the lift-off point (LP) of iso-

therm is shifted to the left when the repeated compression was applied. However, it should be noted that the cycles accompanied by only slight changes in surface pressure indicating a good stability of the floating layers.

The more detailed studies were performed for the QDs floating monolayers on NiCl₂-containing aqueous subphases. It was established that the increase of NiCl₂ concentration in the aqueous subphase (from 1 to 5 mM) leads to the decrease of the gas phase region and the increase of the liquid and condensed phase regions on π -A isotherms.

It was suggested that Ni²⁺ cation interacts with the carboxylic groups of free (unbounded to QDs) oleic acid molecules. As a result, the density of floating layer and the compression module values increased. The highest compression modulus values of QDs floating monolayers were found for the 5 mM NiCl₂ containing subphase.

By studying the floating monolayer stability during 1800 seconds time, it was found that the monolayer area decreased by about 17 % for both concentrations of NiCl₂ (1 and 5 mM). The floating monolayer on the pure water subphase was more stable: the monolayer area was reduced only up to 13.5 %.

The decrease of the subphase temperature from 25 to 19 °C made it possible to increase the stability and uniformity degree of QDs in floating monolayer independently of the subphase composition.

The studied floating monolayers were transferred onto silicon substrate and studied by AFM method. It was found that a rise of the transfer pressure increases roughness of films, regardless the subphase composition. Whereas, the presence of NiCl₂ in the aqueous subphase leads to an increase in the thicknesses and hydrophobicity of the obtained thin films.

The obtained results can be used for the development of multicomponent thin film nanomaterials containing quantum dots.

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