

Structure and properties of Langmuir–Blodgett films based on poly(9-vinylcarbazole) and poly(methyl methacrylate) blends

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Abstract: The relevance of creating coatings based on polymer blends is driven by the challenges of modern materials science, which include obtaining materials with specified performance characteristics by changing the composition of chemical systems and studying their properties. This paper presents the results of a study of the structural, mechanical characteristics and surface wettability of nanostructured films based on polymer blends of polyvinylcarbazole (PVK) and poly(methyl methacrylate) (PMMA). PVK, PMMA and PVK-PMMA films were formed using the Langmuir – Blodgett (LB) method via horizontal deposition onto the pre-hydrophilized silicon substrates. A correlation has been established between atomic force microscopy (AFM) data of the structural, mechanical properties and the wettability of the polymeric coatings. It was shown that at PVK mass content of up to 45 % in the polymer blend solution, the formed LB films are characterized by a disruption of a structural integrity and the formation of spherical and elongated conglomerates. According to static force spectroscopy data, the local values of the elastic modulus and adhesion force of LB films based on PVK-PMMA blends aren't depend on the quantitative ratio of components. The water contacts angle (CA) values for the polymer blends ranged from $74.31^\circ \pm 1.21^\circ$ to $80.11^\circ \pm 1.55^\circ$ and are determined by the properties of the component whose mass content is predominant. The formation of a dense LB film based on a polymer blend with significantly different molecular weights is possible when the low molecular weight component content in the blend is up to 15 wt. %, which is confirmed by AFM data for the PVK-PMMA sample with a component mass ratio is of 87.1 % – 12.9 %. The wettability of this film differs from that one of the homopolymer samples, which expands the application prospects of these polymers.

Keywords: Langmuir – Blodgett method; nanostructured films; polymer blends; poly(9-vinylcarbazole); poly(methyl methacrylate); atomic force microscopy; elastic modulus; wettability.

1. Introduction

Polymer films are promising material which is currently used as sensitive elements in gas (Youssef et al., 2016) and electrochemical (Li et

al., 2024) sensors, components of drug delivery systems (Albarahmieh et al., 2018), and optoelectronic devices (Bilger et al., 2019). However, there remains a need to enhance still problems of challenges remain in expanding the functional characteristics and physicochemical properties of known materials. One approach to solving this problem is the fabrication of film materials based on multicomponent systems: polymer composite materials (Mo et al., 2019; Prakash et al., 2013), polymer blends (Goettler & Scobbo, 2019) and their combinations (Brulé & Flat, 2006).

Researchers are focusing on the development of new materials with the required range of properties based on polymers, because of their wide variety and easy susceptibility to physical and chemical modifications. In (Shahbazi et al., 2016), the changes of the initial material properties were demonstrated by the introduction of various biopolymers into a polyvinyl alcohol (PVA) matrix. It was established that the introduction of k-carrageenan, alginate or chitosan was accompanied by the compaction a denser of the PVA film structure and an increase in its tensile strength values. An increase in the k-carrageenan content in the polymer blend led to a decrease in the wettability of the films. According to Patil et al. (2021), the introduction of PVA into a starch matrix was accompanied by an improvement in the complex mechanical properties of the films compared to the matrix component. The study by Oh et al. (2026) demonstrated that the synergy of properties of the synthesized copolymer poly (glycidyl methacrylate-g-polypropylene glycol)-co-poly(oxyethylene methacrylate) and small amounts of polyactin (25 wt.%) allows for obtaining thin film composite membranes with a semi-interpenetrating network structure with high CO₂/N₂ separation selectivity and minimal adhesion.

A number of studies (Ding et al. 2023; Qin et al., 2026; Li et al.2024) have focused on optimization of the polymer blends composition. It was shown that blending polymers leads to an improvement in the complex of mechanical, optical, and dielectric properties of films.

A wide range of studies have been dedicated to investigating the fundamental aspects of changes in the rheological properties of solutions, as well as the formation of coatings based on polymer blends. Thus, the paper (Xue et al., 2012) showed that the formation of coatings based on polymer blends is generally accompanied by a process of component phase separation as a consequence of their low mutual solubility. Phase separation underlies the structural diversity of multicomponent polymer coatings. It is noted (Kamanyi et al., 2008; Rodríguez-Hernández, 2016) that the film fabrication methods, the conditions of their implementation, the nature and the quantitative ratio of the components of polymer blends influence the phase separation process. Thus, phase separation is a key factor in the structure formation of polymer films, which also determines the material properties.

To form polymer coatings, drop-casting, spin-coating, and Langmuir – Blodgett methods are generally used (Da Rocha Rodrigues et al., 2020). It is known that the LB and spin-coating methods provide a high degree of control over the structure and molecular organization of coatings (Da Rocha Rodrigues et al., 2020; Agarwal et al., 2023). Papers (Mitsui et al., 2012; Kawaguchi, 2017; Torrisi et al., 2010) indicate that the formation of polymer blend films at the air-water interface leads to the “extrusion” of one of the components onto the surface of the monolayer.

The localization of components on the subphase is determined by a combination of their physicochemical properties and their compatibility, including hydrophilic-hydrophobic properties, branching of component molecules, intermolecular interactions, the possibility of forming associates and aggregates, etc. As a result, the structure formation process of films under the LB method conditions is a multi-stage process. Application of the solution to the subphase is accompanied by component restriction and the formation of the thermodynamically equilibrium dense monolayer, which is reflected in the monolayer compression isotherms. The structure of the monolayer is determined by the conditions of the subphase level lowering and the interaction of the monolayer with the substrate surface (Arslanov, 1994).

AFM allows for a comprehensive analysis of the structural and mechanical properties of the resulting monolayer LB films at the nanoscale and reveals the features of their formation at different component contents.

The aim of this work is to form LB films based on PVK-PMMA polymer blends with various quantitative ratios of components and to research their structural, mechanical and hydrophilic properties.

2. Materials and methods

Monolayer polymer coatings were prepared from blends of PVK (Sigma Aldrich, $M = 100\,000$) and PMMA (Sigma Aldrich, $M = 15\,000$). PVK-PMMA solutions with various quantitative ratios of components were used, obtained by mixing PVK solutions ($c = 0.75\text{ mg}\cdot\text{ml}^{-1}$) and PMMA ($c = 1\text{ mg}\cdot\text{ml}^{-1}$) in chloroform. To achieve uniform component distribution, the PVK-PMMA solutions were subjected to ultrasonic treatment for 10 min. As a result, samples of the following compositions (Table 1) were obtained.

Table 1. Qualitative and quantitative composition of samples

Abbreviation	Sample (mass content (%) of corresponding component)
S1	PMMA
S2	PVK-PMMA (7.7-92.3)
S3	PVK-PMMA (24.3-75.7)
S4	PVK-PMMA (42.9-57.1)
S5	PVK-PMMA (63.6-36.4)
S6	PVK-PMMA (87.1-12.9)
S7	PVK

Monolayer polymer films were formed using the LB-method on the surface of silicon substrates (111 orientation) by horizontal deposition using the “Automated complex for modifying membrane surfaces with molecular and ultrathin layers” (A. V. Luikov Heat and Mass Transfer Institute of the National Academy of Sciences of Belarus) (Mel'nikova et al., 2025). PVK-PMMA films were deposited at a surface pressure (π) $15\text{ mN}\cdot\text{m}^{-1}$ (determined on the basis of an analysis of experimentally obtained monolayer compression isotherms in the « $\pi - A$ » coordinates, monolayer compression rate – $0.3\text{ mm}\cdot\text{s}^{-1}$, aqueous subphase temperature – 20°C). Preliminary hydrophilization of silicon substrates was carried out according to the method described in (Sapsalyov et al., 2021).

The monolayer compression modulus (k) was calculated from the linear regions of the polymer blend monolayer compression isotherms corresponding to the “liquid-crystalline film” phase state according to the methodology described in (Sapsalyov et al., 2025).

The structural and mechanical properties of PVK-PMMA films were investigated by AFM using an NT-206 AFM microscope (ALC «Microtestmachines», Belarus) with NSC 11A silicon cantilevers (average stiffness – $3\text{ N}\cdot\text{m}^{-1}$) (Micromash, Estonia) with radius of curvature of no more than 10 nm. The elastic modulus (E) and adhesion force (F) values were calculated based on nanoindentation data (from the “approach-retraction” curves) using Johnson-Kendall-Roberts model (Mel'nikova et al., 2025).

The hydrophilic properties of PVK-PMMA coatings (WCA, specific surface energy) were evaluated on the DSA 100E (Krüss, Germany); working liquids – distilled water, diiodomethane, drop volume – $2\text{ }\mu\text{L}$. The CA was determined by the “lying” drop method (by plotting a tangent curve at the three-phase point: liquid – substrate – air). The specific surface energy was calculated using the Owens – Wendt – Rebel – Kaelble model (Kaelble, 1970).

3. Results

According to the compression isotherms (Figure 1) obtained for solutions with various ratios of polymer components (Table 1) at $\pi = 15 \text{ mN}\cdot\text{m}^{-1}$, the PVK, PMMA and their blend films are in “liquid-crystalline” phase state. The significant increase in surface pressure on the PVK compression isotherm compared to PMMA is caused to the high molecular weight values and the stiffness of the PVK chains.

It should be noted that in the present study, compared to the results presented in (Sapsalyov et al., 2025), the rate of subphase level decrease during LB monolayer deposition onto the silicon substrate was reduced. This extended the time for molecular relaxation and resulted in obtaining the film in a thermodynamically stable phase state.

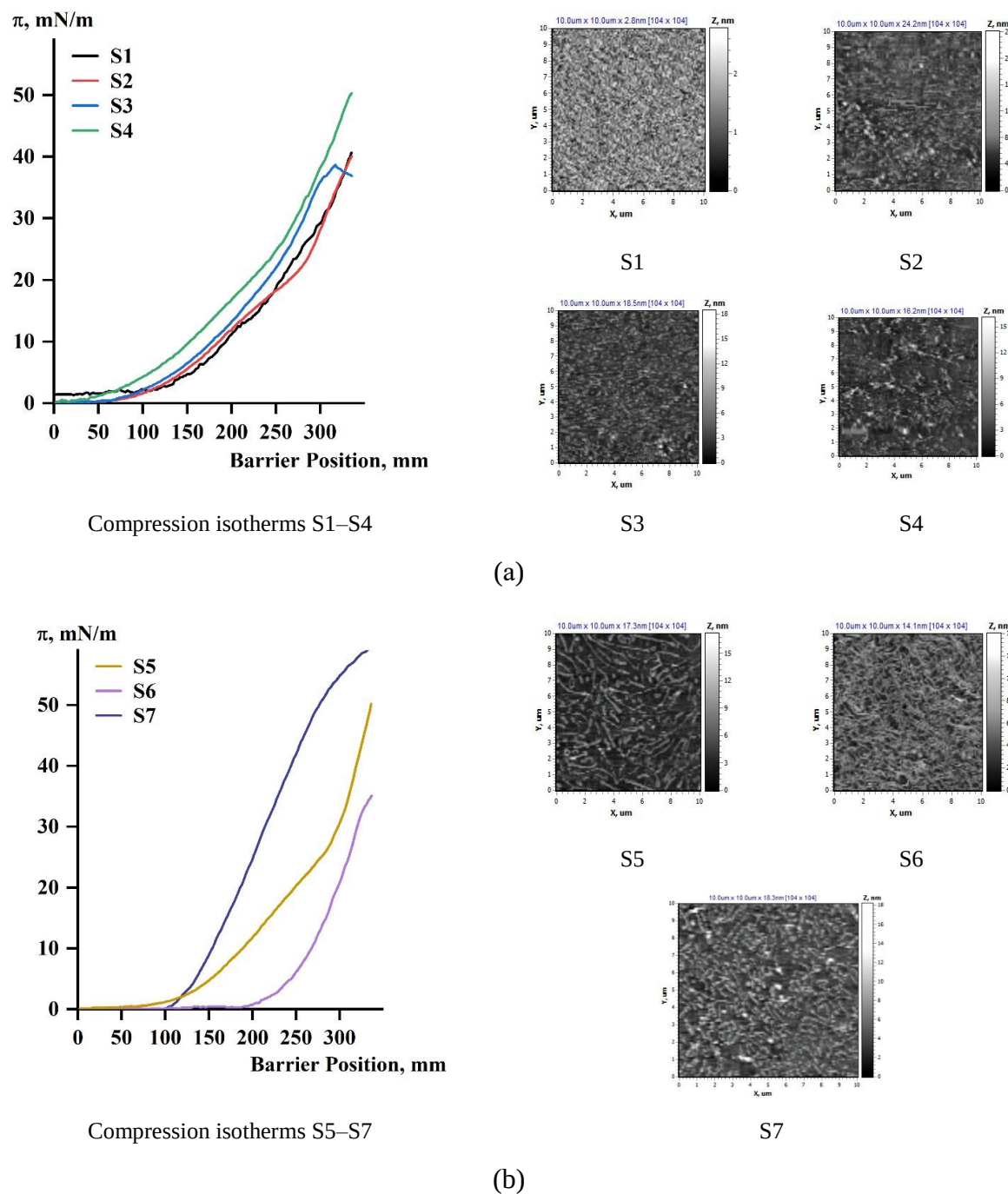


Figure 1. Compression isotherms and AFM structure of LB films: a) samples S1–S4, b) samples S5–S7

Based on the compression isotherms, the compression modulus values of the monolayers made from polymer blends were calculated (Table 2).

Table 2. Compression modulus values of monolayers at different PVK contents

Sample	PVK content, %	Compression modulus (k), $\text{mN}\times\text{m}^{-1}$
S1	0	65.7
S2	7.7	49.6
S3	24.3	69.0
S4	42.9	68.7
S5	63.6	72.8
S6	87.1	114.1
S7	100	93.5 (Sapsalyov et al, 2025)

The formation of films from the solutions with low PVK content (S2) leads to a decrease in k from $65.7 \text{ mN}\cdot\text{m}^{-1}$ to $49.6 \text{ mN}\cdot\text{m}^{-1}$. This pattern of the compression modulus change is explained by the destructuring of the film, which results in the formation of numerous spherical conglomerates, the presence of which is confirmed by AFM data (Figure 1a). The analysis of the shape of the compression isotherms for monolayers based on PVK-PMMA blends (S2–S6) indicates that an increase in PVK content leads to a non-monotonic increase in the tangent of the slope angle of the linear range (an increase in the compression modulus from $49.6 \text{ mN}\times\text{m}^{-1}$ to $114.1 \text{ mN}\times\text{m}^{-1}$) in the corresponding phase region of the « $\pi - A$ » isotherms. The k values for films S3 and S4 are comparable to S1 ($65.7 \text{ mN}\times\text{m}^{-1}$), since PVK conglomerates are localized in the structure and on the surface of the films, while the monolayer is formed by PMMA. The structure of samples S4 and S5 is similar to PVK, which is confirmed by the increase in k from $72.8 \text{ mN}\times\text{m}^{-1}$ to $114.1 \text{ mN}\times\text{m}^{-1}$. It should be noted that k for the sample S6 is higher compared to PVK (Table 2). This fact indicates the formation of a denser structure in the sample S6 through the introduction of PMMA into the intermolecular space of PVK.

Comparison of AFM data showed that the structure of PMMA films is homogeneous and contains a minimal number of defects (Figure 1a). This is caused by the higher segmental mobility of PMMA molecules, which contributes to the formation of dense packing. The PVK structure is characterized by the presence of both large spherical and thread-like elements (Figure 1b), formed because of the lower mobility of polymer segments.

For the S2 film, the introduction of small amounts of PVK into PMMA leads to a disruption of the structural integrity of the films, which is confirmed by a decrease in the compression modulus values of the monolayer (from $65.7 \text{ mN}\times\text{m}^{-1}$ to $49.6 \text{ mN}\times\text{m}^{-1}$). S2 and S3 films are characterized by the presence of numerous spherical conglomerates (Figure 1a), while an increase in the amount of PVK (S4) is accompanied by their growth and the prevalence of irregular spherical shapes. Presumably, the spherical and elongated formations are represented by PVK. The reason for their formation is the low compatibility of the components, accompanied by phase separation.

The surface of film S6 is characterized by a homogeneous filamentous structure. Data on the surface morphology of PVK and S6 show that the introduction of small amounts of PMMA into the PVK solution allows for the formation of uniform films with a structure similar to PVK. PMMA fills the intermolecular space of PVK chains. The increase in the compression modulus of S6 compared

to S7 is attributed to the introduction of PMMA into the intermolecular space of PVK, which leads to an increase in the packing density of the matrix component macromolecules.

Thus, based on the data of the structure and compression modulus values of the studied films, it is shown that the matrix of the monomolecular polymer monolayer is determined by the predominant component in the blend: for samples S2–S4 – PMMA, S6 – PVK. The structure of sample S5 is intermediate compared to S2–S4 and S6. The film of S5 acquires the structure of PVK but its properties are determined by PMMA in terms of the delocalization of PVK conglomerates on the film surface.

According to the results of static force microscopy data (Figure 2), the local elastic modulus values of PVK and PMMA films (S1 and S7) were $412.2 \text{ MPa} \pm 10 \%$ and $507 \text{ MPa} \pm 10 \%$ respectively.

The E and adhesion force values for S2–S6 lie in the range between S1 and S7 and do not change within the confidence interval, which indicates that there is no dependency between the data of mechanical properties of the polymer blend films and the quantitative ratio of the components.

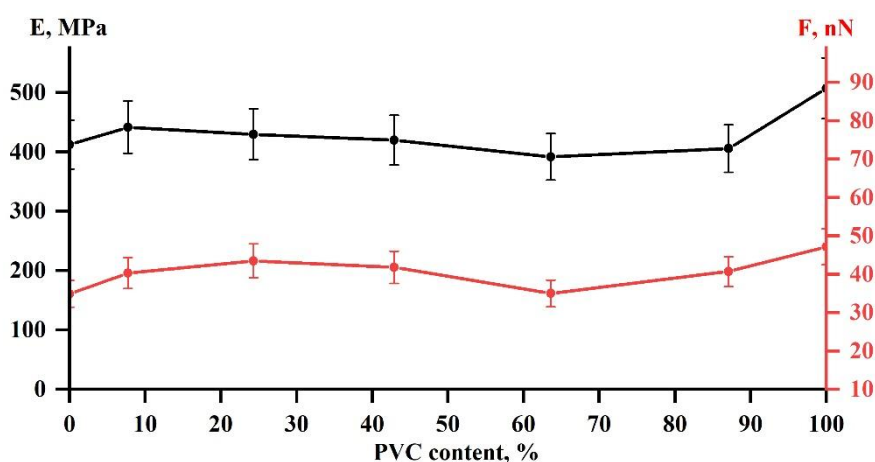
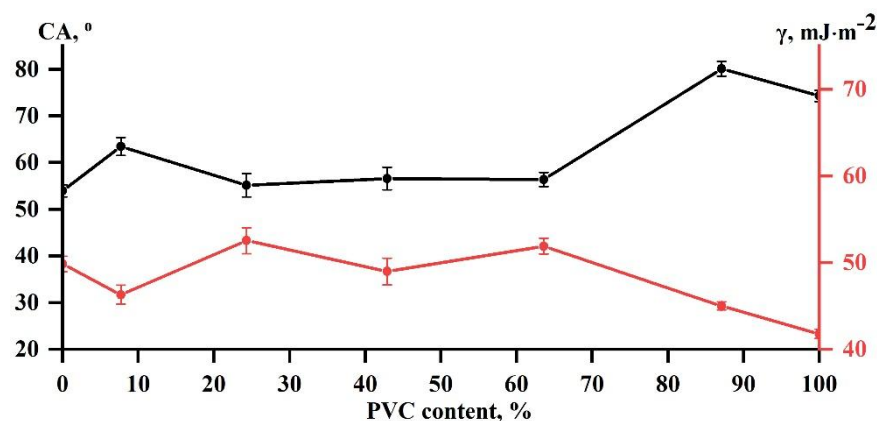


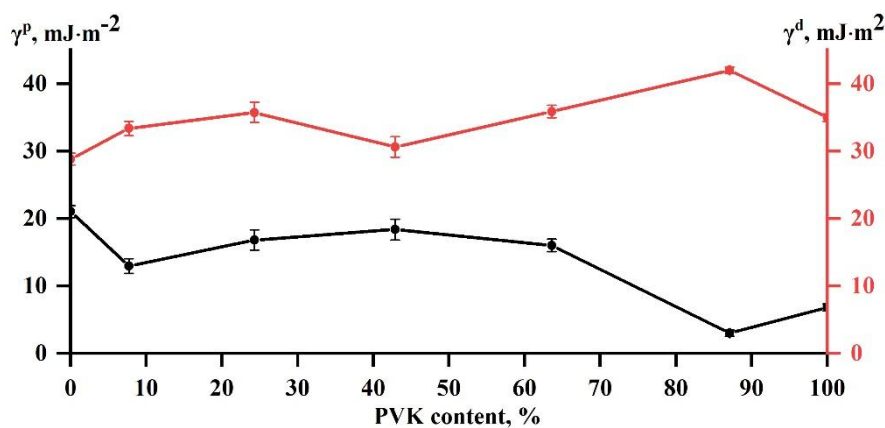
Figure 2. Elastic modulus and adhesion force of PVK-PMMA LB coatings

The CA values of PVK and PMMA films were $74.31^\circ \pm 1.21^\circ$ and $53.97^\circ \pm 1.33^\circ$, which is explained by the difference in the structure of their macromolecules. The macromolecules of PVK and PMMA are composed of polyvinyl chains with carbazole and acetate site groups, respectively. The presence of bulk carbazole groups provides the hydrophobicity of PVK. The acetate groups of PMMA contribute to better wettability of the film surface. The polar component of the specific surface energy for PMMA ($21.03 \text{ mJ} \times \text{m}^{-2}$) is higher than that of PVK ($6.81 \text{ mJ} \times \text{m}^{-2}$). Similar to the changes in the monolayer compression modulus of the films, the specific surface energy values are independent of PVK and PMMA content in the polymer blends (samples S2–S5). The CA and polar component values for films S2–S5 are similar to the S1 sample. In S2–S5 samples PVK is represented by spherical and elongated conglomerates. As a result, the wettability of S2–S5 films is determined by PMMA.

For sample S6, the decrease in the specific surface energy values was established as a consequence of an increase in the dispersion component, and CA values were approximately like PVK (Figure 3). The increase in the CA (from $74.31^\circ \pm 1.21^\circ$ to $80.11^\circ \pm 1.55^\circ$) upon the introduction of small amounts of PMMA into PVK (S6) is attributed to the formation of a denser and more homogeneous filamentous structure.



(a)



(b)

Figure 3. Changes in the CA and specific surface energy (a); changes in the polar and dispersion components of the specific surface energy of LB films based on PVK-PMMA

4. Discussion

Based on research, it was shown that the morphological features of polymer blend films are determined by the structure of the predominant component. The formation of a dense, homogeneous film for PVK-PMMA blends is possible when the matrix is represented by a component with a molecular weight (PVK) significantly higher than the second component (PMMA). The low compatibility of the components leads to the phase separation of the monolayer and the formation of numerous spherical conglomerates and filamentous structures. It is demonstrated that blending PVK and PMMA does not lead to a change in the mechanical properties of the coatings. The elastic modulus and adhesion force of S2–S6 are independent of the quantitative ratio of components. The wettability of PVK-PMMA films is determined by the predominant component.

The obtained results indicate the potential for using PVK-PMMA films as a sensitive layer of electrochemical sensors for analyzing the content of organic impurities and metal ions in aqueous media. In future work, it is planned to develop and investigate the analytical characteristics of capacitive sensors, the sensitive layer of which will be presented by PVK-PMMA films with an optimal component ratio (S6).

5. Conclusion

Thus, for the formation of dense LB films based on polymer blends, the optimal component ratio is PVK-PMMA 87.1 % – 12.9 %, which is confirmed by a complex of AFM data on the structural and morphological features of the film surface, as well as data on surface wettability.

6. Supplementary materials: No supplementary material.

7. Author Contributions

Conceptualization - M.Sh., G.M.; methodology - G.M., S.Ch.; software - S.Ch.; validation - M.Sh., D.S., T.T.; formal analysis - M.Sh.; investigation - M.Sh., G.M., D.Sh.; resources - S.Ch.; data curation - M.Sh.; writing - original draft preparation - M.Sh.; writing - review and editing - M.Sh., G.M., D.S.; visualization - M.Sh.; supervision - G.M.; project administration - G.M., S.Ch. All authors have read and agreed to the published version of the manuscript.

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10. Conflicts of Interest: The authors declare no conflicts of interest.

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Поли(9-винилкарбазол) және полиметилметакрилат қоспалары негізіндегі Ленгмюр – Блоджетт пленкаларының құрылымы мен қасиеттері

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Аңдатпа. Полимер қоспасынан жасалған жабындарды әзірлеу қазіргі заманғы материалтану саласындағы міндеттерге, соның ішінде химиялық жүйелердің құрамын өзгерту және олардың қасиеттерін сипаттау арқылы көрсетілген өнімділік сипаттамалары бар материалдарды өндіруге қатысты. Бұл мақалада поливинилкарбазол (ПВК) және полиметилметакрилат (ПММА) қоспаларына негізделген нанокұрылымды пленкалардың құрылымдық-механикалық қасиеттері мен бетінің ылғалдануын зерттеу нәтижелері ұсынылған. ПВК, ПММА және ПВК-ПММА қоспасынан жасалған пленкалар гидрофилденген кремний пластиналарының бетіне көлденең тұндыру арқылы Ленгмюр – Блоджетт (ЛБ) әдісімен қалыптастырылды. Жабындардың құрылымдық-механикалық қасиеттері мен ылғалдануы туралы атомдық күш

микроскопиясы (АКМ) деректері арасында корреляция анықталды. Полимер қоспасынан жасалған ерітіндіде ПВК мөлшері 45 пайызға дейін болғанда, пайда болған ЛБ пленкалары құрылымдық тұтастықтың бұзылуымен және сфералық және созылған конгломераттардың пайда болуымен сипатталатыны көрсетілген. Статикалық күш спектроскопиясының деректеріне сәйкес, ПВК-ПММА қоспаларына негізделген ЛБ-қабықшаларының серпімділік модулі мен адгезия күшінің жергілікті мәндері компоненттердің сандық қатынасына тәуелді емес. Полимер қоспалары үшін (КУС) мәндері $74,31^\circ \pm 1,21^\circ$ -ден $80,11^\circ \pm 1,55^\circ$ -ке дейін болды және массалық құрамы басым компоненттің қасиеттерімен анықталады. Молекулярлық салмағы айтарлықтай әртүрлі полимер қоспасына негізделген тығыз ЛБ қабықшасының түзілуі қоспадағы төмен молекулалық компонент мөлшері 15 салмақтық %-ға дейін болғанда мүмкін, бұл компоненттік массалық құрамы 87,1 % – 12,9 % болатын ПВК-ПММА үлгісі үшін АКМ деректерімен расталады. Бұл қабықшаның ылғалдануы гомополимер үлгілерінің ылғалдануынан ерекшеленеді, бұл осы полимерлердің қолдану әлеуетін кеңейтеді.

Түйін сөздер: Ленгмюр–Блоджетт әдісі; нанокұрылымды қабықшалар; полимер қоспалары; поли(9-винилкарбазол); поли(метилметакрилат); атомдық-күштік микроскопия; серпімділік модулі; суланғыштық.

Структура и свойства пленок Ленгмюра–Блоджетт на основе смесей поливинилкарбазола и полиметилметакрилата

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Аннотация. Актуальность создания покрытий на основе смесей полимеров обусловлена задачами современного материаловедения, в число которых входит получение материалов с заданными эксплуатационными характеристиками за счет изменения состава химических систем и изучение их свойств. В данной работе представлены результаты исследования структурно-механических характеристик и смачиваемости поверхности наноструктурированных пленок на основе смесей поливинилкарбазола (ПВК) и полиметилметакрилата (ПММА). Пленки ПВК, ПММА и смесей ПВК-ПММА сформированы методом Ленгмюра – Блоджетт (ЛБ) с использованием горизонтального способа осаждения на поверхности гидрофилизированных кремниевых пластин. Установлена корреляция данных атомно-силовой микроскопии (АСМ) о структурно-механических свойствах и смачиваемости покрытий. Показано, что при массовом содержании ПВК до 45 процентов в растворе смеси полимеров для сформированных ЛБ-пленок характерно нарушение структурной целостности и формирование сферических и вытянутых конгломератов. Согласно данным статической силовой спектроскопии, локальные значения модуля упругости и силы адгезии ЛБ-пленок на основе смесей ПВК-ПММА не зависят от количественного соотношения компонентов. Значения КУС для смесей полимеров составили от $74,31^\circ \pm 1,21^\circ$ до $80,11^\circ \pm 1,55^\circ$ и определяются свойствами компонента, массовое содержание которого преобладает. Формирование плотной ЛБ-пленки на основе смеси полимеров, значительно различающимися молекулярными массами, возможно при содержании низкомолекулярного компонента в смеси до 15 масс. %, что подтверждено АСМ-данными для образца ПВК-ПММА с массовым содержанием компонентов 87,1 % – 12,9 %. Смачиваемость данной пленки отличается от образцов гомополимеров, что расширяет перспективы применения данных полимеров.

Ключевые слова: метод Ленгмюра–Блоджетт; наноструктурированные пленки; смеси полимеров; полиметилметакрилат; поливинилкарбазол; атомно-силовая микроскопия; модуль упругости; смачиваемость.