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JSCS–5362 *Review*

REVIEW

Self-assembly of carbon-based nanoparticles films by the Langmuir–Blodgett method

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Abstract: Carbon nanoparticles are a class of materials with extraordinary properties. In the past three decades, four major types of carbon nanoparticles were synthesized and investigated: fullerenes, carbon nanotubes, graphene and carbon quantum dots. One of the main properties of such materials is their hydrophobic nature. At the same time, Langmuir–Blodgett (LB) method the for deposition of thin films of hydrophobic materials provides the possibility to design thin films of different carbon-based nanoparticles with special architectures and features enabling their usage in various fields, particularly in electronics and biomedicine. In this review, the state of art of LB thin films of four types of carbon-based nanoparticles and their application in electronics and biomedicine are presented. The breakthrough in this field was finally achieved by application of carbon quantum dots soluble in solvents optimized for LB deposition.

Keywords: fullerenes; carbon nanotubes; graphene; carbon quantum dots; thin films.

CONTENTS

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1. INTRODUCTION

1.2. Langmuir–Blodgett method for thin films deposition – Procedure and properties

The Langmuir–Blodgett (LB) method is a technique for creating mono- and multi-molecular films by transferring Langmuir (L) films to the surface of a solid substrate (quartz, glass, mica, aluminium, gallium arsenide wafer, silicon wafer, tin and their oxide, silver, gold, *etc*.). The condensed L films are composed of an organized monolayer of amphiphilic molecules floating on water. The heads of amphiphile molecules are dipped into the water whereas the tails stick up into the air. When these molecules spread on water they, remain mostly on the water/air surface. On that surface they can be compressed by movable barriers until the uniform layer is formed (*i.e.*, two-dimensional liquid-like and solid-like arrays), Fig. 1a. By withdrawing a hydrophilic substrate perpendicular to and through the floating monolayer from below, a packed monolayer will coat the substrate (Blodgett introduced this modification), Fig. 1b. Therefore, LB method comprises the transfer of the floating monomolecular layers to solid substrate by vertical dipping it into and out of the bath.[1,2]

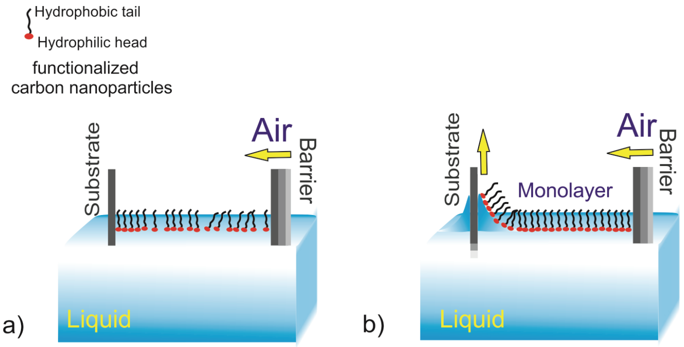

Fig. 1. Schematic view of the deposition technique of Langmuir–Blodgett thin films: a) formation of a floating layer (Langmuir films) on the air/water surface; b) a packed monolayer coat of the substrate by withdrawing a hydrophilic substrate perpendicular to the floating monolayer from below.

LB has being successfully used for the transfer of water insoluble molecules, nanowires, nanoparticles, derivatives of the graphene[3] and transition metal chalcogenides (TMC) materials onto solid substrates. The LB technique uses a special LB trough for nanoparticles injection at the air/water surface. The floating nanoparticles have been compressed closer to each other by using motorized barriers. These barriers allow control of the packing density of the nanoparticles. After that, they are transferred onto the solid substrate by utilization horizontal Langmuir–Schäfer (LS), horizontal lifting (HL) or vertical LB dipping to create a monolayer coating. Controlled coatings can be deposited by multiple-times repeating of the dipping procedure until multilayers of different composition and thickness are obtained.[4] The advantage of the LB method includes a firm control of the film structure and packing density, uniform monolayer deposition over large areas as well as the achieved layer thickness, thus presenting this method to be better and more efficient than other used methods.[5] The LB technique has unique performance that enables two dimensional (2D) functional materials with scalable and controllable growth to be achieved. Various LB parameters, such as speed of immersion and withdrawal of the substrate, composition of mixture of spacer (for example C_{60}) and matrix (for example arachidic acid) molecules, temperature, pH and ionic contents of liquid sub-phase on which the monolayers spread, the change of the deposition scheme, *etc.* can be controlled successfully.[6] LB films usually deposit in a symmetrical mode, referred to as Y-type films (the molecules in successive layers adopt a head-to-head and tail-to-tail arrangement), X-type and Z-type films (deposition occurs only during immersion or emersion of hydrophilic substrate, respectively).[6]

LB films are now often used as the building blocks of molecular electronics, such as optoelectronic and microelectronic devices, bio and chemical sensing, LCDs, lithography, *etc.* They could also be used for creating biological materials for improvement of cell adhesion or to study the properties of biofilms.[7–9] Ideal solvents should have two abilities: to disperse spacer molecules and nanoparticles and to spread on air/water surface. Solvents typically used for LB deposition are water immiscible and good spreading, such as low boiling point chloroform, dichloromethane and cyclopentanone (CPO), or water miscible and good dispersing, such as *N*-methyl-2-pyrrolidone (NMP).[10,11] NMP diluted with chloroform can be used for tailoring the vapour pressure, boiling point and concentration of the spreading dispersion.[10,12] The role of spreading behaviour can be understood from following example. Dichloromethane and toluene are immiscible solvents with very similar surface tensions, γ, of 27.9 and 27.8 mN m^{-1}, respectively. However, the dichloromethane spreads on water whereas the toluene does not.[12,13]

Typical techniques used for characterization of LB thin films are Brewster angle microscopy (BAM), fluorescence imaging microscopy, surface potential measurement, infrared reflection absorption spectroscopy, attenuated total reflection – Fourier transform infrared, ellipsometry, X-ray diffraction/reflection – grazing incidence and small angle X-ray diffraction, neutron diffraction, UV–Vis absorption spectroscopy, Raman spectroscopy, optical harmonic generation, optical microscopy, confocal fluorescence, scanning electron microscopy (SEM), transmission electron microscopy (TEM), scanning tunneling microscopy (STM), atomic force microscopy (AFM), X-ray photoelectron spectroscopy (XPS) or electron spectroscopy for chemical analysis (ESCA) and I-V, C-V resistivity measurements.[14]

1.2. Hydrophobic carbon-based nanoparticles for the deposition of LB thin films

Carbon nanoparticles belong to the class of water insoluble materials that could be deposited by the LB method. They are divided into fullerene, carbon nanotubes, graphene and carbon quantum dots. However, carbon nanoparticles can be modified to become soluble in water. Hydroxylated carbon nanomaterials, such as fullerol $C_{60}(OH)_{24}$ or graphene oxide (GO), are soluble in the water and ethanol.

A fullerene is the carbon allotropic form that has 60 carbon atoms linked by single and double bonds.[15] They are arranged in a spherical shape at the vertices of truncated icosahedron. Fullerenes are commonly produced in plasma reactions among carbon atoms and clusters at temperatures between 4000 and 8000 K.[16–25] Unlike other allotropes, fullerenes are soluble in non-polar solvents, such as toluene, benzene, chlorobenzene, dichloromethane, carbon disulfide and 1,2,3-trichloropropane.[26–28] Fullerenes are practically insoluble in methanol and highly insoluble in water. However, until recently, fullerenes were the only well-known allotrope of carbon that could be dissolved in commonly used solvents at room temperature.[29–33]

In the literature, various applications of C_{60} in nanomedicine, chemistry, biology, electronics, *etc*., are described. The unique physics and chemistry of C_{60} stimulate new research in fundamental and applied science. They are normally electrical insulators. The room temperature resistivity of undoped C_{60} films exposed to air is 10^{14} Ω cm.[34] However, when crystallized with alkali metals, the obtained compound can be conductive or even superconductive.[35] As excellent electron acceptors, C_{60} could be chemically modified with the intention of improving their solubility in organic solvents. Therefore, such derivatives of the C_{60} materials could be one of the best organic n-type semiconductors.[36–38] Furthermore, molecular heterojunctions produced by covalently linking C_{60} with donated electron or photoactive macromolecules could be used as intrinsic p/n-type semiconductors and artificial biological photosynthesis.[39]

However, maybe the most interesting application could be the light transfer to electricity with the possibility of their use for solar cells. In addition, due to their great electric conductivity, C_{60} modified by some inorganic and organic functional groups, non-metallic and metallic elements, could be used for the manufacture of high efficient light emitting diodes (LEDs) or organic light emitting diodes (OLEDs).

Fullerene is typically deposited on a substrate as a film by vacuum evaporation or ion plating.[40–43] One of the characteristic features of fullerenes is their ability to sublimate at considerably lower temperatures as compared to other stable forms of carbon.[44–46] Modification of thin C_{60} films is limited to expensive methods, such as ion beam bombardment.[47–51]

Fullerenes are well-known photosensitizer because they have the ability to generate or quench reactive oxygen species and thus could be used very effectively in photodynamic therapy for the treatment of cancer diseases (skin or eye cancer).[52–57] Fullerene must be photosensitized by UV or visible light to generate highly reactive singlet oxygen or superoxide radicals.[58,59] Derivatized C_{60} fullerene with cationic functional groups forms molecules that are more water-soluble and can mediate photodynamic therapy efficiently upon illumination and they can selectively bind to microbial cells.[60–62]

Carbon nanotubes (CNTs) are another group of the carbon allotropes.[15] This class of carbon nanomaterials are intermediate between graphene (flat sheets) and buckyballs (closed shells). CNTs consist of a hexagonal two-dimensional lattice of carbon atoms joined and bent in one direction. Thus, a hollow cylinder is formed. The structure of an ideal, infinitely long, single wall CNTs (SWCNTs) is the regular hexagonal lattice that has been drawn on the infinite cylindrical surface, the peaks of which present the positions of carbon atoms. The length of the carbon–carbon bonds is pretty fixed and hence in this respect, there are limitations on the diameter of the cylinder and the arrangement of the atoms on it.[63] In terms of elastic modulus and tensile strength, CNTs are the stiffest and strongest discovered materials. This strength results from the covalent sp^2 bonds created between the individual carbon atoms.[64] CNTs are either semiconducting or metallic along the tubular axis.[65–67] Because of the nanoscale cross-section, propagation of electrons is only along the axis of the tube. As a result, CNTs are often called one-dimensional conductors.

CNTs have appropriate properties of photoluminescence (fluorescence), absorption and Raman spectroscopy.[68–74] Spectroscopic methods provide the possibility of the rapid and non-destructive characterization of relatively great amounts of CNTs. Such properties allow reliable and quick "nanotube quality" characterization in term of the structure (chirality) of the produced nanotubes, non-tubular carbon content and structural defects. Such properties define all other properties, such as mechanical, optical and electrical. Due to their apparent hydrophobic nature and strong van der Waals forces,[75] CNTs tend to agglomerate into bundles, which hinder their dispersion in solvents or viscous polymer solutions.[76–79] The obtained nanotube aggregates or bundles obviously reduce the mechanical performances of the finally produced composite. The surface of the CNTs could be modified to reduce the hydrophobic properties and to improve interfacial adhesion to the bulk polymer by chemical attachment.[80–89] Knowledge of physical and chemical characteristics of CNTs, as well as their reactions in organic solvents, play at very important role in the design, production and utilization of their counterparts, which could have improved properties. CNTs derivatives are soluble in organic solvents (*e.g.*, chloroform, cyclohexanone, CPO, acetone and toluene).[90–94]

Graphene presents a single layer of carbon atoms in a two-dimensional hexagonal lattice, in which one atom forms each vertex.[95,96] It is the fundamental structural element of other carbon allotropes, such as charcoal, graphite, fullerenes and carbon nanotubes. Compared to other carbon allotropes, graphene has special and remarkable properties. In proportion to its thickness, graphene is approximately 100 times stronger than other materials, such as the strongest steel. Actually, graphene is the strongest material ever tested.

Large-area graphene films produced by chemical vapour deposition (CVD) are polycrystalline and contain grain boundaries.[97] Grain boundaries with very pronounced disorder are highly frequent in CVD graphene films.[98] Graphene has a Young's modulus of 1 TPa and an intrinsic tensile strength of approximately 130 GPa.[99]

Another method for the production of graphene thin films is rapid thermal annealing of nickel films saturated with carbon atoms, molecules or nanoparticles.[100–103]

In theory, the specific surface area (SSA) of graphene is 2630 m^2 g^{-1}, which is similar to that of activated carbon, but much larger than those of carbon nanotubes (CNTs), from ≈100 to 1000 m^2 g^{-1}, and carbon black (less than 900 m^2 g^{-1}).[104] It conducts electricity and heat very efficiently and is almost transparent.[105] Additionally, graphene shows nonlinear and large diamagnetism, even bigger than graphite, which could be levitated by using Nd–Fe–B magnets.[106] Graphene is a semiconductor with zero bandgap. Its valence and conduction bands touch each other at the Dirac point. Graphene has unique optical properties that produce surprisingly low opacity for atomic monolayers in vacuum, with absorbing $\pi\alpha \approx 2.3$ % of red light (α is the constant of the fine-structure).[107] This property is a result of unexpected low-energy of the graphene monolayer electronic structure that contains hole and electron conical bands.

Soluble fragments of graphene could be prepared in the laboratory by chemical modification of graphite.[108–114] The produced material with circular graphene layers, which has the thickness of the 0.53 nm, is soluble in water, dichloroethane, tetrachloromethane and tetrahydrofuran (THF). Good solvents for graphene, such as CPO, ethanol, chloroform and acetone, are characterized by using Hildebrand solubility parameters: $\delta D \approx (18\ \text{MPa})^{1/2}$, $\delta T \approx (23\ \text{MPa})^{1/2}$, $\delta P \approx$ $\approx (9.3\ \text{MPa})^{1/2}$ and $\delta H \approx (7.7\ \text{MPa})^{1/2}$.[115] Ahmad *et al.* established that graphene dispersed in 2-propanol and THF presented multilayered, wrinkled and overlapping graphene sheets, whereas samples dispersed in NMP and gamma-butyrolactone (GBL) were less thick with predominantly folded graphene sheets.[116] Higher boiling point solvents, such as NMP and GBL, are more effective than 2-propanol and THF in obtaining stable graphene dispersion.

Graphene is normally hydrophobic and the Hummer method is the classical method to make it hydrophilic.[117,118] GO is a single carbon atom layer with functional groups having oxygen attached to the both sides of the flake areas. The addition of oxygen groups results in the breakage of carbon double bonds. In that respect, the material loses its own electrical conductivity. However, these oxygen groups cause that the whole flake to become polar and, consequently, able to disperse in solvents such as water. Since water is a non-reactive solvent, GO dispersions could easily be detached and dried into films, allowing for homogeneous and uniform GO distribution on practically all surfaces.[119–121] GO could be dispersed in NMP, *N*,*N*-dimethylformamide (DMF), ethylene glycol and THF.[122] Full exfoliation of the GO in all these solvents into single-layer GO (SLGO) sheets has been achieved using the sonication process. The GO dispersions showed long-term stability and contained sheets in the range between a few hundred nanometers to a few micrometers, similar to the GO dispersions in water. These results could facilitate the processing of graphene-based materials in different applications.[123] Furthermore, SLGOs with lateral dimensions of 1–20 μm produced by a modified Hummer method are highly dispersable in NMP, deionised water, dichlorbenzene, DMF, and other solvents with behaviour like water.

Electrochemical exfoliation of graphite is a promising alternative to the Hummer method.[124,125] Electrochemically exfoliated sheets are dispersible in water, ethanol, acetone and DMF. Thin films are conductive and do not require reduction.[126]

Carbon quantum dots (CQDs) are produced generally by bottom up methods.[127–131] There are characterized by the following properties: quasi-spherical shape with lateral dimension smaller than 10 nm, amorphous structure-dominantly composed of sp^3 hybridized carbon, good chemical stability, tuneable photoluminescence and optical band gap, resistance to photo-bleaching and low cytotoxicity under ambient light conditions.[132–144] Graphene quantum dots (GQDs) are photoactive nanomaterials produced by a top down method from graphite or carbon nanotubes.[145–149] They have disk-like shape with an average diameter in the range of 2–20 nm and are composed mainly of sp^2 hybridized carbon and as a result, they are more crystalline.[150,151] They are also photoluminescent due to quantum confinement, surface defects and zigzag edges.[152–154] The structure, photoluminescence, band gap or cytotoxicity of CQDs and GQDs can be modified by doping with different elements (nitrogen, sulphur, chlorine, fluorine, *etc*).[155,156] Both CQDs and GQDs can be used as antibacterial/anticancer/autoimmune agents.[157–165] Due to their low dark cytotoxicity, CQDs can also be used for bio-imaging.[166]

2. LB DEPOSITION OF THIN FILMS OF CARBON NANOPARTICLES

2.1. LB deposition of fullerenes

Over the past 30 years, many papers have been published on LB deposition of fullerenes. It was found that LB deposition of a monolayer of fullerene was difficult to achieve without modification. Fullerenes were modified either by usage of matrix and surfactant molecules or by covalent derivatization. The electrical properties of LB layers of modified fullerenes were studied mostly for photovoltaic devices. The parameters of LB deposition strongly depend on the type of fullerene modification.

Nakamura *et al.* described LB fullerene (C_{60}) films formed with or without matrix molecules.[167] They found that collapsed films at the air/water surface were formed by pure C_{60}. The monolayers with icosanoic acid (mixture proportion of 1:1) were transferred onto substrates. In addition, these LB films have a smooth surface. In another research, LB films of pure C_{60} were prepared by using an intermediate phase of an aqueous phenol solution.[168] According to this research, the relatively homogeneous L film formed by C_{60} molecules at the air/water surface was without any matrix molecules. These L films using horizontal lifting method were transferred on the hydrophobic substrate. Xu *et al.* fabricated four species of films, consisting of C_{60}, C_{70} and their mixtures with arachidic acid (AA), onto various substrates by the LB technique.[169] They concluded that by using these materials, condensed layers on the air/water surface could be formed. Berzina *et al.* used the LB technique for deposition of uniform C_{60} films.[170] These authors claimed that the LB films were deposited using a mixture of a specially selected surfactant compound and C_{60}. In the amorphous uniform LB films, the C_{60} molecules and surfactant compound would be distributed homogeneously. Another characteristic of the surfactant compound is its solubility in hexane, in which C_{60} was not dissolved.

Saito *et al.* investigated the photoelectric transient responses in photoelectric cells (Schottky-type) with a photoconductive layer made of a C_{60} and AA mixed LB film.[171] The effects of the symmetry and molecules orientation in existing LB films on the conduction process of photo-carriers was also investigated. Compared to the LB films with merocyanine dye, dispersive conduction was not shown with these films. Only a high speed response that had a time constant in the nanosecond-level or less was shown. These results indicated high-level importance of the symmetry and orientation of molecules in the response. In another research, dispersing poly(3-alkylthiophene)s (P3ATs) with AA as surface-active material, three P3AT–AA–C_{60} mixed systems were produced into Y-type LB films.[172] The transfer ratio was close to unity. The LB films were used for creating Schottky diodes model. After doping with C_{60}, it was found that the P3ATs became semiconductors. The diodes model showed a rectification effect with electronic parameters strongly dependent on the P3AT molecules and their alkyl side chain length. In another experiment, the surface pressures of C_{60} L films were measured as a function of the surface area.[173] According to the expected molecular areas, the engaged area of L films created by a solution of 0.1 mg cm^{-3} was small. The surface morphology of C_{60} LB films transferred onto a mica substrate showed that the particles were aggregated to form flocks. This agreed with the fractal aggregation of crystalline C_{60} islands and explained the smaller engaged areas. A strong attractive interaction among fullerenes as well as steric repulsive interaction among the islands was established. Jin *et al.* investigated L and LB films of two C_{60} derivatives. One with a methoxycarbonyl substituent (C_{60}(I)) and another with a carboxylic group (C_{60}(II)).[174] In addition, their photovoltaic property and the LB films charge-transfer process on n-type Si substrates were studied. Those two C_{60} derivatives LB films showed the same increase on the n-Si direct transition. Furthermore, the structure of C_{60}(II) LB film was investigated using FTIR and X-ray small-angle diffraction. It was detected that such kind of C_{60} derivative created micellar aggregates.[174] In another research, solid porphyrin–fullerene films were produced by evaporating a mixture of fullerene and porphyrin in solution and deposited by the LB method.[175] The dyes were covalently bonded to each other. Both types of films displayed an emission band shifted close to the infrared region (*ca*. 800 nm). The emission was attributed to an emitting charge transfer (CT) state that was formed by fullerene and porphyrin moieties, characterized by a long lifetime (*ca*. 2 ns) and a high quantum yield (0.14 %). Total reorganization energies were deduced from the spectra of the CT emission of the fullerene–porphyrin films.

The capability of a C_{60} derivative to form an L monolayer at the air/water surface was investigated by the surface pressure *vs*. molecular area isotherm as well as by using BAM.[176] The monolayers were transferred onto mica, quartz and ITO substrates using the LB technique. After transfer, they were characterized by AFM and UV–Vis. Conoci *et al.* reported the formation of thin films consisting of the fulleropyrrolidine derivative and a novel water-soluble porphyrin.[177] The LS method was used for their transfer. In particular, a solution of the fulleropyrrolidine derivative in chloroform and dimethyl sulphoxide was spread on the water surface, whereas the novel water-soluble porphyrin was dissolved in the aqueous sub-phase. Results of the UV–Vis spectroscopy showed that the two components behaved as weakly interact discrete π systems. The usage of polarized light indicated that a preferential direction of the macrocyclic rings existed with an edge-on arrangement according to the substrate surface, Fig. 2.

Metzger studied several C_{60} unimolecular rectifiers.[178] It was found that between Au or Al electrodes their molecules showed asymmetric electrical conductivity. Miuira *et al.* prepared a C_{60} film at the air/water surface and transferred onto hydrophobic substrates.[179] In this process, a modified HL method was used for film deposition. The substrates were first submerged before spreading at the air/water surface and then C_{60} film was lifted up. The optical density of the transferred C_{60} LB film was increased 3.9 times by using the modified HL method compared to the conventional HL method.

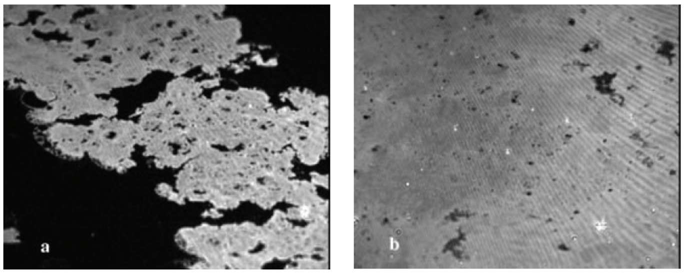

Fig. 2. BAM images of pure fullerene derivate 1 at the air–water surface and a surface pressure of: a) 0.1 and b) 5 mN m^{-1}. The field of view of the BAM instrument along the *x*-axis was 430 μm. Reproduced with permission.[177] Copyright 2003, Elsevier.

AFM observation showed that the C_{60} clusters well covered substrate surface (lateral size 0.1–0.4 μm and in height 30–70 nm), without any areas uncovered. Furthermore, they realized that exposure of the uncovered substrate surface is inevitable by using the conventional HL method. In another research, the fullerene derivative C_{60}TT was substituted with the tris(dodecyloxy) benzamide–low-molecular-weight organogelator.[180] By the LB method, such a derivate created nanowire structures. The holding time before deposition had a strong impact on the C_{60}TT LB film when the pressure on the surface was 5 mN m^{-1}. *Via* a fibrous monolayer structure, it changed the homogeneous monolayer to a bilayer fibrous structure. The estimated dimension was 1.2 nm in height, 5–10 mm in length and 8 nm in width. The holding times of the monolayer fibrous quasi-one-dimensional structures growth was from 0 to 0.2 h. This time was considered as interface-controlled process. The growth time of the bilayer fibrous quasi-one-dimensional structures was from 0.2 to 18 h. It was considered as diffusion-controlled process. Yan *et al.* showed the transfer of the monolayer-high islands of C_{60} and $C_{60}O$ to oxide-terminated Si (111) substrates.[181] Such a transfer was made from L films on phenol sub-phase or L films on water. While both LB multilayers of C_{60} and $C_{60}O$ are granular, significant morphological differences between molecular thin films were observed. In particular, it should be noted that the $C_{60}O$ multilayers had a relatively high density of ring characteristics, with diameters of 100–300 nm, which were not observed in $C_{60(70)}$, Fig. 3.

Furthermore, in another experiment, monolayer LB films of metallofullerene Gd@C_{82} were mixed with stearic acid (Gd@C_{82}/SA).[182] They were deposited on mica substrates from the air/water surface. The results were obtained using AFM and contact angle (CA). The obtained results showed the easy fabrication of metallofullerene LB films with SA (Gd@C_{82}/SA) by using the vertical dipping method, which was not the case with the pure metallofullerene film. It was found that compatible LB films have an ordered and uniform layer structure when the molar ratio of the SA:Gd@C_{82} was up to 4:1. Koulompis *et al.* described a new bottom-up approach by combining self-assembly with the LS deposition technique.[183] As a result, a graphene-based layered hybrid material was synthesized with fullerene molecules within the interlayer space. Thus, preparation of films consisted of the bottom-up layer-by-layer process that was provided *via* the creation of hybrid organo-graphene oxide L films. Such films were deposited on hydrophobic substrates. Amer and Altalebi were the first to use the LB technique for successful production of hexagonal close-packed monolayer films from unmodified C_{60} fullerene.[184] Under optimized processing parameters, the structure of the films was investigated using STM and AFM. Such films could be used in many applications such as optoelectronic devices, transparent electrodes, sensors and photovoltaics.

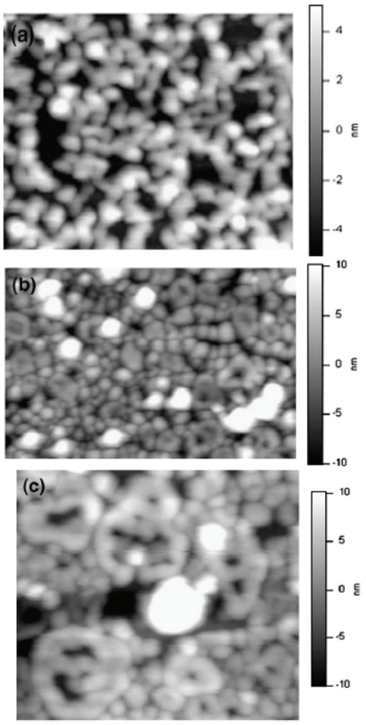

Fig. 3. a) AFM image (0.8×1.2 μm^2) of a C_{60} multilayer L film transferred at a target pressure of 10 mN m^{-1} to an oxide-terminated Si (111) surface. The concentration and volume of the C_{60} in benzene solution were 5×10^{-5} M and 300 μl, respectively; b) AFM image (1.0×1.5 μm^2) of $C_{60}O$ multilayer on oxide-terminated Si(111) (4×10^{-5} M solution of $C_{60}O$ in toluene; total volume 400 μl; target pressure 10 mN m^{-1}); c) Higher resolution (1.3×1.4 μm^2) of "doughnut"-shaped $C_{60}O$ aggregates. Reproduced with permission.[181] Copyright 2009, Elsevier.

2.2. LB deposition of carbon nanotubes

Since the discovery of CNTs in 1991, many studies have been conducted regarding the nanotube layers deposited by the LB technique. They faced basic problem of incompatibility of CNTs with solvents preferred for LB deposition. Various modification procedures of CNTs had to be perf0rmed in order to prepare them for LB deposition. Unlike fullerenes, up to 99 layers of parallel arrays of modified CNTs could be easily deposited. Basic electrical phenomena of LB CNT films were investigated and potential applications immediately emerged, mostly in nanoelectronics. The starting quality of CNTs, their method of production, separation and modification are major parameters for successful deposition of LB films.

It was found that composites of CNTs incorporated in polymer matrixes showed high importance for developing materials with increased mechanical strength and for optical applications. Krstić *et al.* presented NTs embedded in LB films as a suitable model to explore the phase behaviour of these materials.[185] SWCNTs have been dispersed in the water, followed with purification based on centrifugation.[185] The resulting dispersions were then spread into a sub-phase polyelectrolyte. They found that crucial problem for controlling manipulation of SWCNTs were their damage-free purification, which could only be achieved by using methods with surfactant solution.[186–187] It was found that purification techniques in organic solvents did not improve the CNT quality.[188] Since researchers failed to produce tube dispersions in an organic solvent suitable for the use of a standard LB technique, they employed different approaches. In this respect, it was presented how such a problem could be solved through a suitable sub-phase composition, in these cases a stabilized water soluble tube/surfactant complex at the air/water surface.[185] In another study, L films consisting of SWCNTs and surfactant molecules were deposited on substrates with lithographically defined electrode arrays.[189] AFM revealed a matrix structure of the LB monolayers which was different compared to the monolayers on bare substrates. Measurements of electrical transport were performed on a single thin bundle at 4.2 K and room temperature. Under low-temperature conditions, the characteristics current–voltage had a non-linear dependence (steps-like features). In order to produce adequate SWCNTs dispersion for LB, Guo *et al.* proposed a new processing technique of SWCNTs.[190] Actually, it was multi-layer LB films in which solubilised SWNTs were dispersed in an amphiphilic polymer matrix, spread on the water surface and deposited vertically on the substrates. The optical absorbance at 1820 nm was perfectly proportional to the number of layers. Thus, layer-by-layer growth of the SWCNTs thin films was confirmed. Furthermore, Raman spectra and polarized absorption indicated some degree of tube orientation in the dipping direction. Although the stability of the LB film was somewhat lowered, deposition of the films without a polymer matrix was possible. In this way, the production of the SWCNTs thin films with optical transparency and precisely controlled thicknesses could be important and a significant breakthrough for understanding and technologically applying CNTs.

In another study, the deposition of optically homogeneous thin films of SWCNTs was realized using the LB technique.[191] Deposition process was performed layer-by-layer (up to 25 layers), allowing for very precise film thickness control. Absorption spectra of such LB films preserved all essential spectral features that are specific for semiconducting and metallic SWCNTs. It was proven that their electronic structures did not sustain serious changes during functionalization and chemical treatment. In addition, recorded AFM profiles of SWCNTs monolayers indicated that most of the tubes were either isolated or existed in small bundles with a size of 1–6 nm.[191] Penza *et al.* presented the optical and acoustic sensors coated by nanotubes, which were successfully studied for sensing applications of volatile organic compound (VOC) at room temperature.[192] They found that LB films composed of tangled bundles of SWCNTs are transferred on different converting sensors. This was realised by linker-buffer LB multilayered material with cadmium arachidate, which was pre-deposited on the sensor surface for better adhesion of the SWCNTs. During the experiment, the hybrid system was successfully recognized, as well as data fusion improvement from optical and acoustic sensors by using highly discriminating SWCNT-functionalized sensors. In other research, monolayers of CNTs–cytochrome c (Cyt c) conjugates were stabilized at the air/water surface.[193] SWCNTs as well as the multiwall CNTs (MWCNTs) have been used for the preparation of conjugate monolayers. By using the LB method, (CNTs–)Cyt c monolayers were transferred onto electrode surfaces. The results of the electrochemical measurements of the (CNTs–)Cyt c electrodes deposited by the LB technique indicated Cyt c reversible redox waves. Compared to a pure Cyt c LB film, the intensity of the redox current was increased for the CNTs–Cyt c conjugate electrodes modified by a LB film. In this way, apart from supporting the creation of ultrathin Cyt c LB films, CNTs can also improve the electron transfer between electrode and proteins. Li *et al.* reported that SWCNTs showed advanced properties desirable for applications in high-performance nanoelectronics.[194] They realized that for future production of high-current, high-speed and high-density nanotube circuits, a large-scale SWCNTs assembly in densely aligned forms could be important. Guo *et al.* reported that by using a parallel nanotubes array as the transistor channel, parasitic capacitance per tube could be reduced.[195] A large improvement in the high-frequency performance can be achieved by increasing the tube density. Moreover, they found that despite progress in assembly and oriented synthesis including the LB method,[196–202] there is no method for the production of pristine SWCNTs assemblies without extensive covalent modifications, high density and high extent of SWCNTs alignment. They developed a new LB method for deposition of monolayers of aligned non-covalently functionalized SWCNTs from organic-based solvents with high-dense packing, Fig. 4. For different bulk materials of various diameters, this method has been presented as generic.[194]

Giancane *et al.* described LB deposition as a technique for producing films with essential control and opportunity for modulation using thickness and molecular organization.[203] They also presented the preparation, characterization, and application of LB films pristine or functionalized (single or multiwall) CNTs in different kinds of research. Venet *et al.* prepared thin films containing SWCNTs by the LB technique and investigated the film morphology by AFM.[204] Pure SWCNTs films were prepared as a network of interconnected tubes bundles and their in-plane electrical characteristics were measured at room temperature. It was established that for low applied voltages, the pure SWCNTs layers exhibited relatively high ohmic conductivity, while non-linear current voltage behaviour was exhibited in the mixed LB films. In this way, high power dissipation could lead to reorganization of the nanotubes in the films with pure nanotubes. Massey *et al. also* produced transparent, thin, electrically conductive films containing pristine SWCNTs by the LB method.[205]

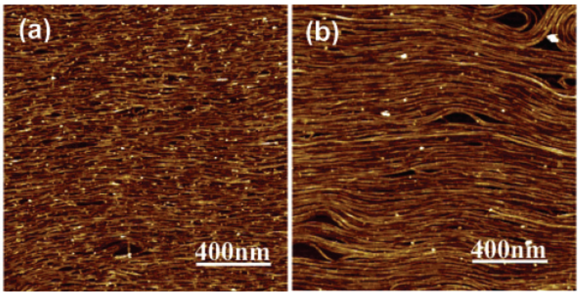

Fig. 4. LB monolayers of aligned SWCNTs: a) AFM image of a LB film of Hipco SWCNTs on a SiO_2 substrate; b) AFM image of a LB film of laser-ablated SWCNTs. Reproduced with permission.[194] Copyright 2007, Journal of the American Chemical Society.

In this way, films of up to 99 layers (thickness 300 nm) could easily be built-up. Electrical and optical measurements detected anisotropy in the film plane, which suggested alignment of the tubes in the deposition process. For most of the films, measurements of DC conductivity in the range 77–300 K showed increasing conductivity with increasing temperature (semi-conducting characteristics). However, it was found that some samples showed the opposite effect. For example, metallic films were reverted to semi-conducting after an electrical annealing process. In another of Massey's research, LB deposition was used to create thin film networks of semiconducting and metallic SWCNTs, Fig. 5.[206]

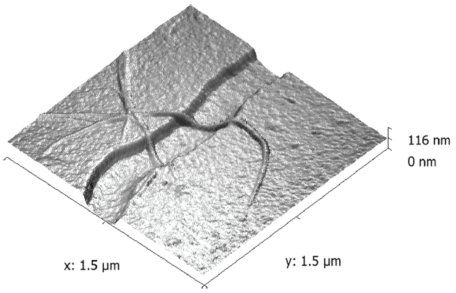

Fig. 5. AFM image (tapping mode) of a single LB layer of SWCNT-S showing two bundles of SWCNT-S bridging a pair of palladium electrodes on glass. Reproduced with permission.[206] Copyright 2012, Journal of American Chemical Society.

These systems were investigated to understand their optical, physical and morphological properties. The electrical conductivities were measured in the temperature range from 80 to 350 K for electrode gaps of 220 and 2 mm. Investigation of semiconducting CNTs showed that the Poole–Frenkel conduction model was the dominant electrical process in the electric fields stronger than 1 MV m^{-1} and temperatures below 150 K.

On the other hand, metallic nanotube networks showed a decrease in resistance with decreasing temperature. This dependence could be linearly approximated by giving the temperature coefficient of resistance, specifically 10^{-3} K^{-1}. In another research, the transparent conductive films (TCFs) composed of functionalized SWCNTs and ultra-large graphene oxide (UL–GO) were deposited on a flexible poly(ethylene terephthalate) (PET) substrate using the LB technique.[207] After reduction by hydriodic (HI) acid, the TCFs provided excellent electrical and optical properties with an outstanding sheet resistance. At a transmittance of 90.3 %, the sheet resistance was 8.1 kΩ sq^{-1}. It was also observed that great improvement of the PET hydrophilicity could be made by deposition of an extra layer of a hydrophilic coating onto the PET substrate. This was mainly due to the introduced functional hydrophilic groups, as well as the high roughness of the surface of the coating layer. It was found that such a highly efficient approach to self-assembly could be used to produce TCF devices that would not have large-scale post-transfer processes. In addition, Kędzierski *et al.* reported that randomly dispersed CNTs thin films are highly promising material for producing transparent electrodes, Fig. 6.[208]

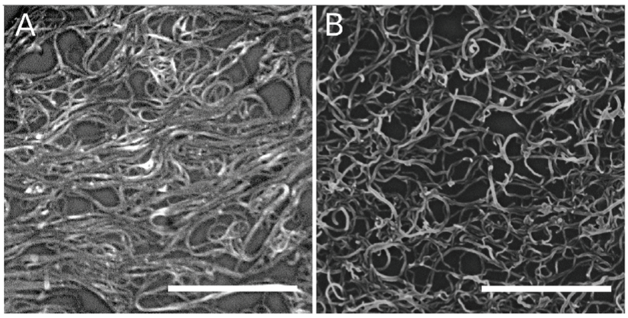

Fig. 6. a) SEM images of SWCNTs and b) MWCNTs thin films; scale bar 1 μm. Reproduced with permission.[208] Copyright 2017, Elsevier.

It was concluded that understanding and knowing the nature and characters of the conductivity of the films are crucial for improvement of their electrical properties. In this respect, the electrical conductivity was investigated of the SWCNTs and multi wall CNTs (MWCNTs) thin films that had been deposited on a polymeric substrate using the LS technique. The conductivity of the films was consistent with the Variable Range Hopping (VRH) model. Moreover, remarkable differences in the conductivity of SWCNT and MWCNT films were found. In addition, the pertinent impact of annealing thin films and their temperatures history on the conductivity performances were realised. Research of CNTs layers transferred on polymeric substrate was performed with the view of possible applications of the films in transparent flexible electrodes. The SWCNTs have metallic and semiconducting properties that are dependent on their chirality. Thus, controlling the electrical properties of SWCNT-based devices is challenging.

Nam *et al.* presented a monolayer SWCNT bundle LB film that exhibited anisotropic electrical properties due to the aligned assembly, Fig. 7.[209]

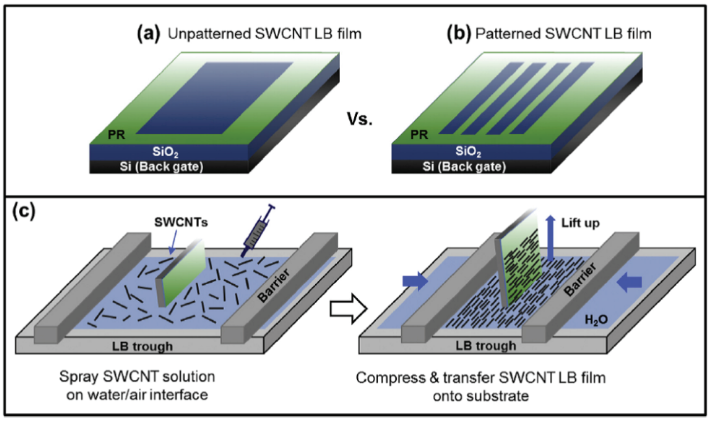

Fig. 7. Schematic illustration of the procedure of aligned SWCNT LB film using a combination of both the LB method and photolithography. Reproduced with permission.[209] Copyright 2019, Elsevier.

Therefore, the devices produced from this film indicated different gating effects according to the channel dimension. The channel dimension was adjusted by a photolithography procedure and the target area of the substrate was where the LB film was deposited. It was also realized that modulation of the semiconducting behaviour of devices based on SWCNTs was significantly affected by control of the channel dimension. They found that the metallic pathway was drastically decreased by shrinking the channel dimensions, which in turn enhanced the semiconducting properties of the device.

2.3. LB deposition of graphene

Graphene and its modifications can also be deposited on various substrates by the LB method. Researchers mostly used graphene and GO dispersed in solvents that provide good exfoliation. Since these solvents are miscible with water, they are not optimized for LB deposition. Several deposition parameters have to be simultaneously varied in order to obtain mono or multilayer films with difficulties. Basic charge transfer phenomena and potential applications were mostly studied.

Li *et al.* prepared graphene films by the LB technique applying the layer-by-layer model.[210] The intention was to explore the opportunity for developing and production of scalable devices based on chemically performed high quality graphene sheets. They found that the process of exfoliation–intercalation–expansion of the graphite could produce high-quality single-layer graphene sheets that are long term suspended in some organic solvents. In addition, the graphene sheets showed high electrical conductivity at cryogenic and room temperatures. In another research, SLGO was presented as a soft and unconventional type of material that could be used for the production of electronic and composite applications.[211] The authors reported how atomically thin graphene sheets were assembled. They found two fundamental geometries of how single layers could interact: face-to-face and edge-to-edge. Such interactions were explored at the air–water interface by LB assembly. The stable monolayers of SLGO were obtained without the use of any surfactant or stabilizing agent. This was realised due to the existence of strong electrostatic repulsion among the 2D confined layers (100). Cao *et al.* presented a new class of molecular field-effect transistors with high-performance and photo-responsive characteristics.[212] Such transistors were formed by LB monolayers of copper phthalocyanine (CuPc). The two-dimensional and ballistically-conductive single-layer graphene was used for planar contacts. They concluded that a unique feature was the merging of LB techniques with the production of the nano-gap electrodes for building functional molecular electronic devices. They found that LB techniques were reliable and promising methods for the preparation of large area ordered ultrathin films with precise and well-defined architectures. In another research, the monolayer GO sheets with dimension of up to ≈200 μm were synthesized using a chemical method and fractionated to obtain four different grades with uniform dimensions.[213] Conductive transparent films of ultra large GO (UL–GO) sheets were produced by the LB assembly technique. The degrees of wrinkling and density of the UL–GO monolayers were tuned by varying the LB processing conditions. They changed from dilute and close-packed flat UL–GO to GO wrinkles (GOWs) and concentrated GO wrinkles (CGOWs). It was found that high-yield fabrication of CGOWs or GOWs could be considered as promising materials for supercapacitors, hydrogen storage, and nanomechanical devices. Park *et al.* presented simple cost-effective intercalation that was based on the exfoliation method.[214] They used it for preparing high-quality graphene flakes, which established stable dispersions in organic solvents without any surfactant. It was confirmed by using XPS and X-ray diffraction that alkali metal intercalation between graphite interlayers through diffusion of the liquid-state from ternary KCl–NaCl–$ZnCl_2$ eutectic system was efficient and successful. In their experiment, a modified LB method was used. The results indicated that this method could be a scalable and economical way for the synthesis of non-oxidized graphene flakes.

In another research, new methods for the development and production of large sized GO with a controllable structure were demonstrated.[215] Such a structure is the key for the unique creation of the soft building block of GO dispersions for the production of electronic devices. In this method, ultra-large GO (UL–GO) sheets (lateral size up to ≈100 µm) are produced in aqueous media and then deposited on substrates with a controllable structure, including standing collapsed, rippled, flat, folded and over folded, scrolled and highly curved morphologies, *via* the LB technique. Using MD simulations, it was found that the GO was much softer than the sheets of pristine graphene. It was concluded that this was due to the sp^3-hybridization produced by oxygen functional groups. Yang *et al.* reported the usage of reduced GO (rGO) LB films as the hole injection layer with high performances in organic light-emitting diodes (OLED).[216] It was found that using the LB technique, thickness-controlled and well-ordered rGO sheets were incorporated between conducting transparent indium tin oxide (ITO) and active organic layer leading to an increase of recombination between holes and electrons. Due to the dramatic jump in the efficiency of hole carrier injection in the rGO LB layer, the luminance performance of the device was greatly enhanced compared to devices produced by spin-coating rGO and the conducting commercial polymer PEDOT:PSS, as a layer for transportation of holes. The results showed that an excellent substitute for the commercial PEDO T:PSS could be an rGO LB film as the efficient electron blocking and hole transport layer in LED devices. Han *et al.* reported a study on a photoluminescence (PL) near-UV blue in GO films that was prepared by an LB-based method, Fig. 8.[217] GO films were reduced into rGO by thermal process at 800 K. The surface morphology of GO and rGO were characterized by scanning electron microscope (SEM) and AFM. XPS results showed significant restoration of the graphitic

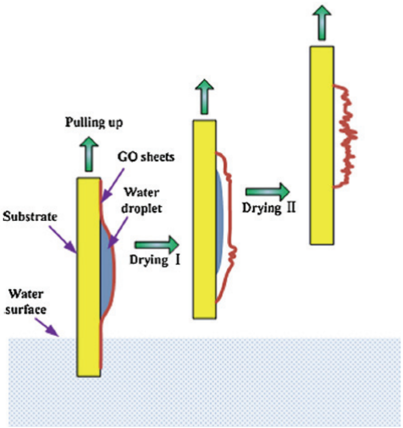

Fig. 8. Schematic view of the transfer and thermal processes of rGO films. Drying I is the drying process in air overnight. Drying II is the thermal treatment at 800 °C under Ar protection. Reproduced with permission.[217] Copyright 2015, Elsevier.

domains in GO. In addition, the PL of rGO exposed the emission of the blue near-UV. The blue shift and decrease in PL were mostly due to the newly created sp^2 clusters in rGO, which were the established pathways of percolation between the already presented sp^2 clusters.

The quality of LB GO and rGO thin films depends significantly on the presence of water molecules on the GO sheets and substrates. Namely, after the GO films were pulled out from water at a low speed and initially dried in air, the GO with a lateral dimension smaller than 5 μm allowed for the fast evaporation water. In this way, the GO sheets did not wrinkle. Water droplets are very often trapped between the GO sheets and the substrate, causing wrinkling of these sheets due to capillary forces. Nevertheless, thermal treatment was the main reason for the wrinkling of GO sheets due to removal of water droplets and oxygenated functional groups.[217]

Matković *et al.* showed a facile production technique for highly transparent and conductive thin graphene films.[218] Sheet conductivity of the multi-layer graphene films assembled by using LB was improved through doping with nitric acid. The transparency remained the same as that of undoped films. Encapsulated films have a long term improved sheet conductivity effect. The stacking of multiple layers and the dependence of the sheet resistivity on axial strain were also investigated. In another research, the wrinkling and folding of monolayer GO using the LB technique were also demonstrated.[219] First, they deposited cetyltrimethylammonium bromide (CTAB) and GO on the air/water surface and uniaxial compressed to form a monolayer. The CTAB enhanced the in-plane rigidity of the monolayer through hydrophobic tail aggregation, which prevented GO–GO in-plane sliding behaviour. They found that overcompression of the GO monolayer resulted in out-of-plane periodic nanoscale wrinkling, which in turn, generated folds that were stable during deposition onto a substrate and chemical reduction of GO. In addition, they investigated a potential application of this material by constructing a 3D electrode of the arranged nanofolded GO–CTAB layers, which exhibited superior volumetric capacitance compared to those of commercial devices. The high volumetric capacitance was attributed to electrolyte-accessible channels that were generated by nanofolds with a dimension similar that of the hydrated ions. Seliverstova *et al.* presented results of the optical and structural properties of SLGO LB films in dependence on temperature.[220] They found that heating of SLGO films over 100 °C leads to a decrease in the optical density of the samples, but the film structure was not changed. Using Raman spectroscopy, they observed a partial reduction of the disordered and highly defective GO in the LB films, followed by ordering of amorphous sp^3--regions and their transition to sp^2-bonded carbon clusters. It was also noticed that the thickness of the LB films decreased. In another research, was investigated the assembly of the film of silica-encapsulated, colloidal semiconducting quantum dots, deposited by the LB method and inserted between two graphene sheets,[221] The luminescent and electrically insulating film was used as a dielectric, with the top graphene sheet sampled in the electrode and successfully utilized like the top gate for the underlying graphene field-effect transistor (FET). It was also found that such heterostructures allowed for the development of the new hybrid optoelectronic devices through the integration of 2D and 0D materials. Dzhanabekova *et al.* presented the effect of the transfer pressure on the optical, structural and electrical properties of LB rGO films.[222] In their research, the rGO monolayer predominantly existed in the liquid state. In the case of increased surface pressure on the film, it was found that the rGO particles were closer in space and contacted one another, which was followed by an overlapping and crumpling of some film sheets. Furthermore, electron microscope studies showed that when monolayers were transferred on solid substrates, the rGO films form uniform and island-type films with clearly observable clusters. It was found that their density grew with increasing surface pressure. The synthesized rGO films were highly transparent (87–96 %) in the visible spectral region. In addition, at a lower surface pressures, LB films transferred onto firm substrates exhibited the best conductive and physical properties. It was realized that the results obtained during the research could be used in the development of a new technology applicable for synthesizing nano-sized transparent films from rGO and its derivatives, which could be used in photovoltaics and molecular electronics.

2.4. LB deposition of carbon quantum dots

There are only few reports related to LB deposition of CQDs. Mostly, hydrophilic CQDs used with graphene sheets to created hybrid structures. Great difficulties were experienced in the formation of monolayers due to the hydrophilic nature of the starting materials. A breakthrough in the field was the design of hydrophobic CQDs soluble in chloroform. This type of CQDs can be deposited in LB monolayer films in a reproducible and cost efficient way.

Kouloumpis *et al.* prepared graphene/carbon dot hybrid thin films by a modified LS method.[223] GO nanosheets were used as templates for grafting C-dots in a bi-dimensional array. Repeating the cycle is a facile and low-cost layer-by-layer procedure for the formation of highly ordered hybrid multilayers. Park *et al.* fabricated a closely packed GQDs film (GQDF) from colloidal solutions using a solvent-assisted LB method, and investigated the optical and electrical characteristics of the heterostacked graphene/GQD film (G/GQDF) structures.[224] It was observed that the GQDF played a role not only as a buffer layer that isolates chemical vapour deposited graphene (CVD graphene) from undesired p-doping but also as a photoactive layer that triggers n-doping of the heterostacked CVD graphene film. Wang *et al.* used an LB technique to fabricate ultrathin, high-quality GQD aggregated films with well-modulated optical properties in a wide range of wavelengths.[225] Through the combination of a bottom-up synthesis of GQDs and the LB assembly method, uniform, closely packed, and ultrathin GQD films could be self-assembled with a well-controlled thickness on different substrates. Stanković *et al.* prepared uniform and homogeneous CQDs thin films using the LB technique.[226] The composition of the CQDs was tuned so that a highly concentred and stable colloid of CQDs in chloroform was produced. These authors investigated the possible antibacterial activity of CQDs films against *S. aureus* and *E. coli*. It was found that LB CQDs thin films had different effects on *S. aureus* and *E. coli*. Namely, *E. coli* is more sensitive to the surface of LB hCQDs thin films compared to *S. aureus*. The CQDs produce singlet oxygen under blue light. The produced singlet oxygen diffuses from LB CQDs films and kills bacteria. Possible mechanism of antibacterial activity of LB CQDs thin films is presented in Fig. 9. It is supposed singlet oxygen attacked the membrane wall, damaged it by increasing its porosity and allowing radical products to reach the cytoplasmic membrane causing its lipid peroxidation.[227]

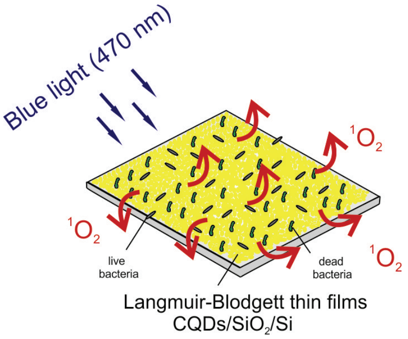

Fig. 9. Mechanism of antibacterial activity of LB CQDs thin films.

CONCLUSIONS

This review provided, for the first time, a comprehensive report on LB thin films of carbon-based nanoparticles with their possible applications in nanoelectronics and biomedicine. A key problem remains identical to 30 years ago. Any modification of carbon nanoparticles that provides their solubility in a desired solvent for LB deposition deteriorates their electronic and optical properties. These properties of carbon nanoparticles are preserved better if a non-covalent approach is chosen. Since CQDs were the first carbon nanoparticles produced by low temperature non-plasma methods, their chemical composition can be tuned to provide solubility in LB preferred solvents. Thus, new perspectives in LB deposition of carbon nanoparticles are opened.

Vacuum methods for deposition of carbon nanoparticle films preserve more of their preferred properties. However, the principal advantage of the LB method is the price of the final product, which is two order of magnitude lower compared to the cost of vacuum deposited thin films.

Any breakthrough in carbon nanoparticles modification without loss of their electronic and optical properties will allow immediate upgrade of LB technology to industrial level and large scale application of carbon nanoparticle coatings for various applications.

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ИЗВОД

САМОУРЕЂИВАЊЕ ТАНКИХ СЛОЈЕВА УГЉЕНИЧНИХ НАНОЧЕСТИЦА ЛАНГМИР–БЛОЏЕТ ПОСТУПКОМ

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Угљеничне наночестице спадају у групу материјала са изузетним особинама. У последње три деценије су испитиване особине 4 главне врсте угљеничних наночестица: фулерена, угљеничних нанотуба, графена и угљеничних квантних тачака. По својој природи ови материјали су хидрофобни. У исто време, Лангмир–Блоџет поступак за депоновање танких слојева хидрофобних материјала омогућава дизајнирање танких слојева ових материјала са особинама које омогућавају њихову примену у различитим областима, посебно у електроници и биомедицини. У овој ревији је дат приказ особина танких слојева 4 врсте угљеничних наночестица депонованих Лангмир–Блоџет методом и њихова потенцијална примена у електроници и биомедицини. Значајан напредак у области је остварен применом угљеничних квантних тачки које су растворне у растварачима погодним за Лангмир–Блоџет депоновање.

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