

VILNIUS UNIVERSITY
CENTER FOR PHYSICAL SCIENCES AND TECHNOLOGY

BRONĖ LENKEVIČIŪTĖ-VASILIAUSKIENĖ

ORGANIC LIGHT EMITTING DIODES: THE INVESTIGATION OF NEW
MATERIALS AND STRUCTURES TOWARDS BROAD EMISSION SPECTRA

Summary of doctoral dissertation
Technology Sciences, Material Engineering (08 T)

Vilnius, 2016

The research has been carried out in 2012–2016 at the Department of Solid State Electronics, Vilnius University.

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BRONĖ LENKEVIČIŪTĖ-VASILIAUSKIENĖ

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PAIEŠKA PLAČIAM REGIMOSIOS SPINDULIUOTĖS SPEKTRUI GAUTI

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Santrauka

1987 metais pasirodė organinių junginių sluoksnių, pasižyminčių elektros srovės sukelta liuminescencija ir pavadintų OLED (*ang.* Organic Light Emitting Diode, OLED) prietaisais [1]. Šie palyginti pigiai ir greitai gaminami šviesą emituojantys organiniai diodai atvėrė naują mokslinių tyrimų sritį. Šiuo metu OLED plačiai taikomi plonasluoksnių lanksčių ekranų rinkoje bei sparčiai veržiasi į bendrojo apšvietimo gyvenamosioms patalpoms rinką: 2014 metais įmonė „Konika Minolta“ sukūrė ir pradėjo masiškai gaminti lanksčius plataus regimosios spinduliuotės spektro OLED prietaisus, kurių šviesinis efektyvumas siekia 131 lm/W [2, 3].

Tačiau vis dar susiduriama su problemomis – kol kas OLED prietaisai nepasižymi dideliu emisinių sluoksnių spektriniu stabilumu, mėlynoje regimojo spektro srityje spinduliavimo efektyvumas didinamas pasitelkiant TADF (*angl.* TADF – thermally activated delayed fluorescence) spinduliavimą [4], taip pat ieškoma kuo paprastesnės OLED sluoksnių struktūros plataus spektro regimajai spinduliuotei gauti.

Šiame darbe buvo tirti VU Chemijos fakulteto mokslininkų sintetinti benzochinolino, naftochinolino dariniai su chloro, tetrazolo, iminofosforano, amino pakaitais, 1,8-naftiridino dariniai su biciklo[3,3,1]nonano, biciklo[3,3,0]oktano, kamparo pakaitais bei jų panaudos perspektyvos plataus spektro OLED spinduliuotei gauti. Rezultatai pristatomi darbuose [5] ir [6]. Taip pat, siekiant padidinti plataus spektro OLED emisinių sluoksnių stabilumą tirta hibridinė struktūra, kuomet spinduliuotė gaunama iš organinės medžiagos $\text{Ir}(\text{Fppy})_3$ ir iš $\text{CdS}_x\text{Se}_{1-x}/\text{ZnS}$ kvantinių taškų. Rezultatai pristatyti darbe [7].

Summary

In 1987 new organic electroluminescent compounds appeared. The layers of these compounds exhibited luminescence properties under current excitation [1] and they were named Organic Light Emitting Diodes (OLEDs). These comparatively cheap and quickly produced in terms of fabrication organic light emitting devices have opened new research area for scientists. Nowadays OLEDs are used in the flexible screen market and are rapidly engaging towards general illumination market. In 2014, “Konica Minolta” began mass production of flexible broad spectra OLED based sheets with the luminous efficiency of 131 lm/W for general lighting [2, 3].

But OLEDs are suffering from spectral stability problems thus far. The efficiency of blue emitters was highly improved using TADF (Thermally Activated Delayed Fluorescence) emitters [4]. Also optimal and requiring less technological steps in terms of price OLED structure is in search for broad spectra illumination.

In this dissertation we are going to present benzo- and naphthoquinoline derivatives (based on already known synthons) with different substituents: chlorine, tetrazole, iminophosphorane, amine; and 1,8-naphthyridine based compounds having a non-planar bicyclic moiety: bicyclo[3.3.1]nonane, bicyclo[3.3.0]octane and camphor which were synthesized at the Faculty of Chemistry, Vilnius university. Also we are going to present these materials as potential emitters in multi-layered OLED systems. All results are published in corresponding research papers: [5] and [6]. In addition, to improve broad spectra stability the hybrid OLEDs (QD OLEDs) with Ir(Fppy)₃ and CdS_xSe_{1-x}/ZnS quantum dots (QDs) as emissive layers (EmL) were shown. The results are published in research paper [7].

Work goals and objectives of the dissertation covers research, characterization of quinolone and 1,8-naphthyridine derivatives. Also fabrication, characterization of OLEDs and hybrid QD OLEDs (whose emissive layers are based on quinoline, 1,8-naphthyridine compounds and Ir(Fppy)₃/CdS_xSe_{1-x}/ZnS QDs respectively) are shown.

Small molecular weight quinoline derivatives are used as fluorescent dyes or as emissive, charge transport layers in OLED devices. Some of synthesized quinoline derivatives are published earlier [8, 9, 10, 11, 12]. *The first aim* of this work was to investigate luminescence properties of the newly synthesized quinoline derivatives with

chlorine, tetrazole, iminophosphorane, amine substituents for broad spectra OLEDs applications.

Also 1,8-naphthyridine derivatives were synthesized. As mentioned previously in the literature [13, 14, 15] 1,8-naphthyridine compounds are used as hole, electron transport and emissive layers in OLEDs. Still, emissive layers with 1,8-naphthyridine derivatives showed concentration dependent photoluminescence (*PL*) quenching and unstable thin film formation. To overcome these issues nonplanar bicyclic moieties (bicyclo[3.3.1]nonane, bicyclo[3.3.0]octane and camphor) were attached. And *the second aim* of this work was to investigate luminescence properties of these newly synthesized 1,8-naphthyridine compounds for broad spectra OLEDs applications.

The third aim of this work was to extend OLED spectra utilizing $\text{CdS}_x\text{Se}_{1-x}/\text{ZnS}$ quantum dots. It is worth to notice that few organic emissive layers giving broad emission spectra in OLEDs degrade differently in the time scale and their emission spectrum depends on applied external voltage. Since quantum dots (QDs) are stable, they may partially solve the colour shift problem and prolong device lifetime. QDs have narrow emission spectra [16] which do not depend on applied voltage. Herein, we report broad spectrum hybrid quantum dot organic light emitting diodes (QD OLEDs) based on two emission layers (EmL): blue-green small organic molecule emitter $\text{Ir}(\text{Fppy})_3$ which does not show any colour shift and red emitter made from core-shell type $\text{CdS}_x\text{Se}_{1-x}/\text{ZnS}$ QDs when concentration of these QDs solution is changed.

Main objectives

1. To investigate how different substituents: chlorine, tetrazole, iminophosphorane, amine in quinoline derivatives and different substituents: bicyclo[3.3.1]nonane, bicyclo[3.3.0]octane and camphor in 1,8-naphthyridine derivatives change their *PL* and absorbance spectra, the values of quantum yields (*QY*) and ionization potential values.
2. To demonstrate experimentally how different substituents used as emissive layer of quinoline and 1,8-naphthyridine derivatives change electroluminescence (*EL*) spectra of OLEDs.
3. To determine a chemical compatibility between quinoline, 1,8-naphthyridine derivatives and $\text{CdS}_x\text{Se}_{1-x}/\text{ZnS}$ QDs.

4. To show and to optimize in terms of concentration of QDs broad spectra hybrid OLEDs utilizing emissive layers made from organic material Ir(Fppy)₃ and from CdS_xSe_{1-x}/ZnS QDs.

Novelty

All chinoline and 1,8-naphtyridine derivatives with different substituents as well as OLEDs based on emissive layers made from these compounds and hybrid OLEDs based on material Ir(Fppy)₃ and on CdS_xSe_{1-x}/ZnS QDs, described in these thesis, are new and have not been reported before.

Main new results.

1. Measured values of chinoline derivatives of the HOMO, the LUMO_{opt} energy levels and optical band gap E_{g_opt} were in range $E_{HOMO}=[-5,9; -5,5]$ eV, $E_{LUMO}=[-3,6; -3]$ eV, $E_{g_opt}=[2; 2,8]$ eV. The difference between *PL* spectra peaks in the solutions and in the layers of the same materials $\Delta\lambda_{PL}=[-42, 84]$ nm. The *PL* solvatochromaticity of quinoline derivatives in toluene, chloroform and 2-methoxyethanol is $\Delta\lambda=[12,77]$ nm. The value of E_{g_opt} energy increases in all quinoline groups while substituents are changed as follows: amine, tetrazole, iminophosphorane and chlorine. Additional benzene rings in the molecules lowered E_{g_opt} energy value and shifted *PL* and absorbance spectra peaks towards shorter wavelengths. The *QY* value in chloroform solution reached 49% while (3c) material was used. The *QY* values of the other quinoline derivatives in chloroform solution were between 0,5% and 17%. The *QY* values of the organic layers did not exceed 10% respectively.

2. OLEDs with structure ITO/TPD/EmL/TmPyPB/Alq₃/LiF/Al showed *EL* spectra in blue and green region when (2c), (2d), (2e), (2f) and (3c), (5b), (5f) chinoline derivatives were used in the emissive layer. When (3d), (4b), (4c), (4f) and (5g) derivatives were used as EmL in ITO/TPD/EmL/TmPyPB/Alq₃/LiF/Al OLEDs the broad visible spectra light were obtained due to *EL* component originated from TPD material.

3. Measured values of 1.8-naphtyridine derivatives of the HOMO, the LUMO_{opt} energy levels and optical band gap E_{g_opt} were in range $E_{HOMO}=[-5,9; -5,4]$ eV, $E_{LUMO}=[-3,4; -2,4]$ eV, $E_{g_opt}=[2,3; 3]$ eV. The bathochromic shift between *PL* spectra peaks in the solutions and in the layers of the same materials was observed. (18), (36) and

(37) 1,8-naphthyridine derivatives had the longest π conjugation in the group and *PL QY* in the layer of these materials reached 16%, 4% and 11% respectively.

4. OLEDs with structure ITO/TPD/EmL/TmPyPB/Alq₃/LiF/Al showed *EL* spectra from red-orange to blue region when 1,8-naphthyridine derivatives: (15), (32), (35), (30), (28), (34), (31), (36), (17), (18), (26), (27), (33) and (29) were used in the EmL. When duplex non-planar “V” conformation molecules ((16), (19) and (21)) were used as EmL in ITO/TPD/EmL/TmPyPB/Alq₃/LiF/Al OLEDs the broad visible spectra light were measured. The white light of these OLEDs could be originated from emission of emissive layer as well as emission of TPD and TPD/EmL interlayer.

5. Chinoline, 1,8-naphthyridine derivatives and CdS_xSe_{1-x}/ZnS QDs in toluene are not chemically compatible because toluene dissolves chinoline and 1,8-naphthyridine derivatives. For that reason hybrid OLEDs were made using Ir(Fppy)₃ material which is chemically compatible with the CdS_xSe_{1-x}/ZnS QDs dissolved in toluene.

6. We have fabricated broad spectra light-emitting diodes based on organic material Ir(Fppy)₃ emissive layer and CdS_xSe_{1-x} /ZnS QDs emissive layer with different concentrations of QDs. The optimal concentration of QDs in toluene is 5mg/ml. The wide EL emission properties of Ir(Fppy)₃ and the simultaneous existence of incomplete energy transfer from Ir(Fppy)₃ to CdS_xSe_{1-x} /ZnS QDs and carrier trapping directly on CdS_xSe_{1-x} /ZnS QDs lead to broad light emission in visible spectrum. This is the simplest component and structure in the realization of broad spectrum QD OLEDs. Our results show that an organic and inorganic hybrid with proper concentration as the active medium could be a promising route to broad spectra QD OLEDs.

Statements to defend

1. The origin of broad visual spectra in OLEDs based on EmL of quinoline derivatives: (3d), (4b), (4c), (4f) and (5g); is the emission from these chinoline derivatives and TPD.

2. The origin of broad visual spectra in OLEDs based on EmL of duplex non-planar “V” conformation 1,8-naphthyridine derivatives is the emission from emitters, from TPD and from TPD/EmL interlayers.

3. The wide EL emission properties of Ir(Fppy)₃ and the simultaneous existence of incomplete energy transfer from Ir(Fppy)₃ to CdS_xSe_{1-x} /ZnS QDs and carrier trapping

directly on CdS_xSe_{1-x} /ZnS QDs lead to broad light emission in visible spectrum in ITO/TPD/Ir(Fppy)₃/QD/Alq₃/LiF/Al structure.

Layout of the thesis

The dissertation consists of 7 Chapters. The first chapter is **Preface**. In the **second chapter** we will find the history of light sources, the goals and objectives of this work, novelty, new results, and statements to defend. In addition, the articles and conference presentations concluding the presented data are listed at the **second chapter**. In this chapter the theory of small organic molecules and quantum dots as well as the basic structures of OLED and of hybrid QD OLED are presented. In the **third chapter** we will find information about fabrication technology of the OLEDs and QD OLEDs described in these thesis. The experimental techniques and the images of microscopes, *PL*, *QY*, ionization potential results of quinoline, 1,8-naphtyridine derivatives as well as of QD OLEDs are presented in the **fourth chapter**. Original *EL* spectra results of fabricated OLEDs with chinoline, 1,8-naphtyridine compounds and of QD OLEDs are presented in the **fifth chapter**. The main conclusions and results of these theses are presented in the **sixth chapter**. The **seventh chapter** consists of the list of references cited in this work.

4.2. HOMO and LUMO values of the emissive layers

In order to optimize energetically the functional layers of OLEDs the HOMO and LUMO values should be evaluated. All HOMO and LUMO, Fermi levels of material layers used in our OLEDs are known from literature [17, 18, 19, 20] and detailed in **Table 2**.

Table 2. The energy values for OLED functional layers

Material	HOMO (ε_v), eV	LUMO (ε_c), eV	ε_F , eV
ITO	-	-	-4,7
LiF	-14	-1	-
Al	-	-	-4,3
TPD	-5,4	-2,1	-
TmPyPB	-6,7	-2,7	-
Alq ₃	-5,8	-3,1	-
CdS _x Se _{1-x} /ZnS quantum dots	-6,7±0,1	-4,7±0,1	-
Ir(Fppy) ₃	-5,5	-3,3±0,5	-

HOMO and LUMO energy values for emissive layers were measured using UV photoelectron spectroscopy in the air method (described in subsection 4.2.1.) and using optical absorption spectra (described in subsection 4.2.2.) respectively.

4.2.2. Absorbption spectroscopy

The values of energy level of the LUMO_{opt} were calculated from the difference of values of the HOMO energy level and E_{g_opt} . The HOMO and the LUMO_{opt} energy level values are presented in energy diagrams of **Fig. 41** and **Fig. 42** (in respect of 0 eV energy vacuum level). It is important to notice that LUMO_{opt} value can vary about 0.5 eV from LUMO value of the material [21].

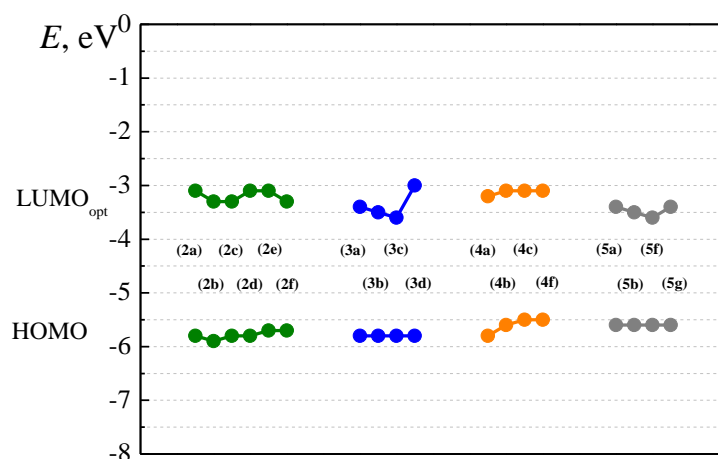


Fig. 41 The energy diagram (values of the HOMO and the LUMO_{opt} energy levels with respect to 0 eV vacuum level) of quinoline derivatives with chlorine-group 2, amine-group 3, iminophosphorane-group 4 and tetrazole-group 5 substituents [5]

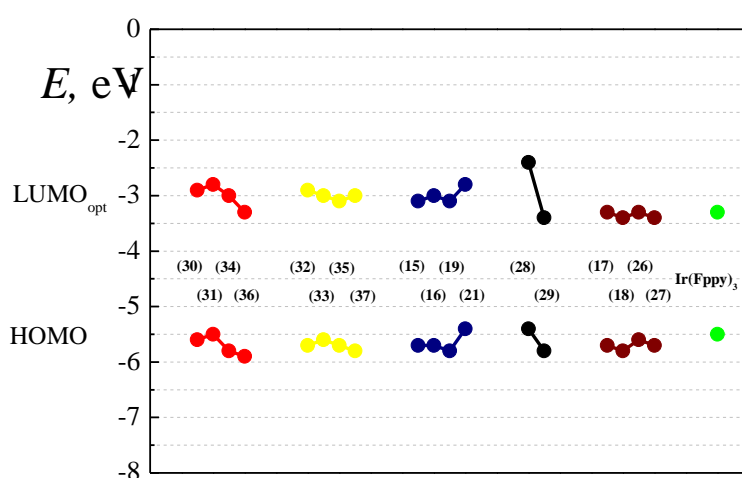


Fig. 41 The energy diagram (values of the HOMO and the LUMO_{opt} energy levels with respect to 0 eV vacuum level) of 1,8-naphthyridine derivatives with dioxy (materials: (30), (31), (34), (36)), dithio (materials: (32), (33), (35), (37)), duplex (materials: (15), (16), (19), (21)), camphour (materials: (28), (29)), oxo (materials: (17), (18), (26), (27)) substituents [6] and of Ir(Fppy)₃ material [7]

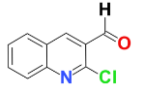
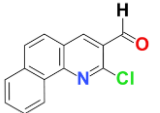
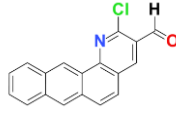
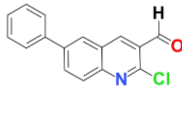
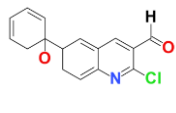
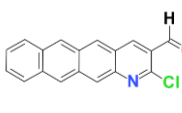
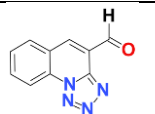
4. 3. Photoluminescence

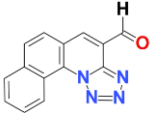
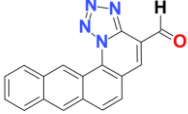
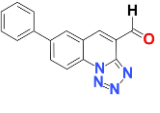
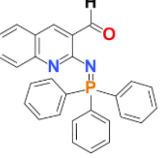
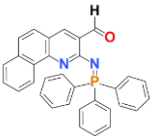
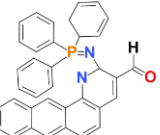
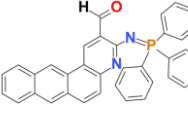
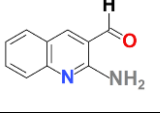
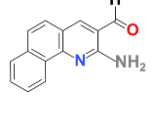
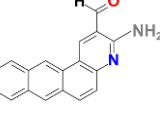
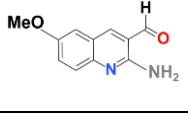
Continuing the material investigation, photoluminescence spectra of materials as thin films and as low concentration solutions were captured. Quantum yield (*QY*) of *PL* in solution and in the thin films was measured under 380 nm wavelength illumination for selected materials. All measurements for chinoline and naphthyridine derivatives are given in subsections 4.3.1. and 4.3.2. respectively.

The *PL* spectra for hybrid QD OLEDs are given in subsection 4.3.3.

4.3.1. Photoluminescence properties of quinoline derivatives

Table 3. Characteristics of photoluminescence and optical absorption [5]

Code	Chemical structure	$\lambda_{solution}^{PL}$, (QY, %)	λ_{layer}^{PL} , nm (QY, %)	$\Delta\lambda$, nm	$\lambda_{layer}^{absorbance}$, nm	$\Delta\lambda'$, nm
(2a)		479;546 (1)	479; 549 (0,01)	0; 3	378	101
(2b)		480 (1)	477; 493 (0,02)	-3	408	69
(2c)		487 (6)	545; 505 (1)	58	395	110
(2d)		483 (2)	–	–	380	–
(2e)		481; 535 (1)	490; 542 (0,01)	9; 7	405	85
(2f)		485; 533 (12)	569 (0,5)	84	454	116
(3a)		471 (0,5)	–	–	349	–

(3b)		485 (1)	555 (0,01)	70	391	164
(3c)		536 (49)	596; 542 (5)	60	436	106
(3d)		476 (2)	–	–	325	–
(4a)		534; 498 (8)	492; 562 (0,01)	-42	420	72
(4b)		515; 531 (16)	569 (5)	54	453	116
(4c)		531 (15)	559 (4)	28	460	99
(4f)		532; 517 (12)	564 (3)	32	458	106
(5a)		486; 528 (0,8)	556 (0,01)	70	402	154
(5b)		485 (23)	539; 505 (10)	54	438	67
(5f)		493 (21)	561 (5)	68	412	149
(5g)		537; 503 (6)	563 (4)	26	424	139

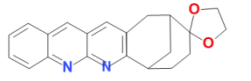
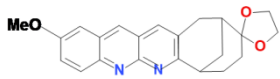
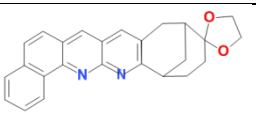
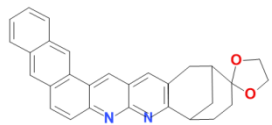
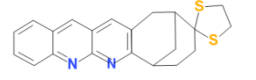
The $\lambda_{solution}^{PL}$ represents the highest intensity maximum. Chloroform used as a solvent. λ_{layer}^{PL} value of the longest wavelength UV absorption peak corresponds to the shortest wavelength PL maximum (to be of the same electronic transition). Comparing to

PL in solution, in most cases *PL* of thin film had a bathochromic shift (given in **Table 3**). This is a natural behaviour of organic molecules [22] proving that in a form of low concentration solution and in the thin layer molecules can form J and H aggregates, can interact through intermolecular interactions and so forth. The biggest difference between *PL* maxima of solution and layer $\Delta\lambda$ was found in material (**2f**) with a value of 84 nm most likely because of better packaging properties in thin film influenced by relatively flat molecular structure. Material (**4a**) showed interesting behaviour to have a considerable negative $\Delta\lambda$ -40 nm shift.

The values of *PL QY* of thin emissive layer should be high enough for OLED applications. The highest *QY* values of thin film chinoline derivative layers were registered for materials (**3c**), (**4b**), (**5b**) and (**5f**). The *PL* properties of the others chinoline derivatives were poor.

4.3.2. Photoluminescence properties of 1,8-naphthyridine derivatives

Table 4. Characteristics of photoluminescence and optical absorption [6]

Medžiagos kodas	Chemical structure	E_{g_opt} , eV	$\lambda_{solution}^{PL}$, nm (<i>QY</i> , %)	λ_{layer}^{PL} , nm (<i>QY</i> , %)	$\Delta\lambda$, nm
(30)		2,7	418; 436; 442 (1)	-	-
(31)		2,7	490; 450 (8)	532; 521; 550 (0.5)	42
(34)		2,8	409; 430 (18)	520; 547;494 (1.2)	111
(36)		2,6	448; 473 (49)	490; 569 (6)	42
(32)		2,8	419; 439; 444; 399 (2)	-	-

(33)		2,6	451; 475 (9)	519; 551 (0.05)	68
(35)		2,6	410; 431 (21)	452; 483;529; 438 (2)	42
(37)		2,8	449; 474 (53)	594; 628 (14)	145
(15)		2,6	439; 460 (2)	-	-
(16)		2,7	450; 470 (25)	-	-
(19)		2,7	409; 431 (20)	489; 532; 438 (8)	80
(21)		2,6	417; 437 (20)	487; 550 (4.5)	70
(28)		3	404; 417; 422 (1)	490 (0.05)	86
(29)		2,4	397; 418; 508 (28)	540; 490 (2)	143
(17)		2,4	415; 436 (22)	-	-
(18)		2,4	454; 479; 510 (50)	480; 530 (11)	26
(26)		2,3	451; 474 (33)	576 (5)	125
(27)		2,3	450; 474 (30)	560;497 (5)	90

As we have a π -conjugated system, two peaks in the *PL* spectra of the solution should correspond to two vibronic states. *PL* spectra of the smallest molecules from each group ((**30**), (**32**), (**15**), (**28**) and (**17**)) are shifted to the blue side compared to the others because of the shortest conjugation length. Naturally, every additional benzene ring in a molecular structure (e.g. comparing compounds (**30**), (**34**) and (**36**)) shifts the spectrum towards the longer wavelengths direction. Spectral characteristics and *PL QY* values are summarized in **Error! Reference source not found.**. Further analysis of the results let us draw some important inferences about structural influence on spectral properties of *PL*. Firstly, an electron donor Ar-O-Me group causes a red shift in *PL*. Having look at all spectral maximums, material (**29**) case appears unique because of a rise of the third wide peak at 508 nm, which could originate from formation of the aggregates. Further investigation is needed to ascertain the nature of this spectral mystery. Comparing to *PL* in solution, in all cases *PL* of thin film had a bathochromic shift. The causes of these discrepancies in *PL* spectra could be few: aggregation of the molecules, crystallization, isomerisation, migration and so forth.

Relatively high ~ 50 % photoluminescence quantum yield was measured for molecules ((**18**), (**36**) and (**37**)) with the longest π -conjugated system in their group. *PL* quantum yield values clearly increase with the extension of π -conjugated chain. But thin layers of (**36**), (**37**) and (**18**) materials gave several times poorer values of *PL QY*: 16%, 4% or 11% respectively (**Table 4**). So the nonradiative relaxation is more intensive in the thin films comparing them to solution of the same materials.

The results of investigation of *PL* properties suggest that materials ((**18**), (**36**) and (**37**)) could be used as blue fluorescent dyes due to quite high *PL QY*. And it is not unlikely that further extension of conjugated chain may increase *PL* efficiency even more.

4.3.2. Photoluminescence properties of QD OLEDs

For better understanding of the QD OLED performance, the energy levels of the HOMO and LUMO for all layers used in the device should be known. The energy levels diagram of hybrid QD OLED is drawn in **Fig. 53**.

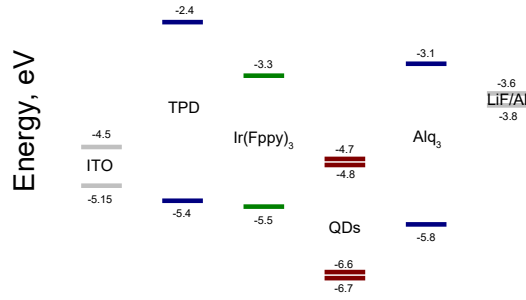


Fig. 53. The band diagram of the QD OLED [7]

As it can be seen from **Fig. 53.** that there is 0.7 eV barrier from anode ITO to TPD, 0.15 eV barrier from TPD to Ir(Fppy)₃ and ~ 1.25 eV barrier from Ir(Fppy)₃ to CdS_xSe_{1-x}/ZnS QDs for holes. While the electrons should overcome ~ 0.5 eV barrier between Al/LiF electrode and Alq₃, then the electrons could be captured by CdS_xSe_{1-x}/ZnS QDs or could move to Ir(Fppy)₃ layer (because it is not known if QDs form monolayer in the structure) and form excitons in Ir(Fppy)₃ layer. These excitons can recombine emitting light in blue-green visible light region or move during Förster's transfer to QDs [23]. The absorption coefficient of thin layer of QDs is ~ 1.5% in the similar systems [24] so irradiative transfer from Ir(Fppy)₃ layer to the layer of QDs is not much possible. Also we could not observe any *PL* of single thin QD layers made from different concentration toluene solutions when we excited these layers with UV light. Förster's energy transfer is a long-range resonant process governed by the dipole–dipole coupling between the donor (in this case Ir(Fppy)₃) exciton and the future acceptor (in this case QD) exciton. If we want that Förster's energy transfer took place between the donors and the acceptors in our QD OLEDs, *PL* relaxation times of phosphorescent organic materials should be on the order of 0.5–10 μs, which are significantly longer than the *PL* relaxation times of QDs [25]. Also the donor (Ir(Fppy)₃) *PL* spectrum has to overlap with the acceptor (QDs) absorption spectrum [24]. In our case all the requirements are fulfilled. Also we can have emission from Alq₃ layer.

Figure 54 shows normalized *PL* spectra for OLED with structure ITO/TPD/Ir(Fppy)₃/Alq₃/LiF/Al (device #1) and QD OLEDs with the structure

ITO/TPD/Ir(Fppy)₃/QD/Alq₃/LiF/Al when QD thin layer is made from different concentrations of QDs in toluene solution: 3 mg/ml, 5 mg/ml, 10 mg/ml and 20 mg/ml (devices #2, #3, #4 and #5 respectively).

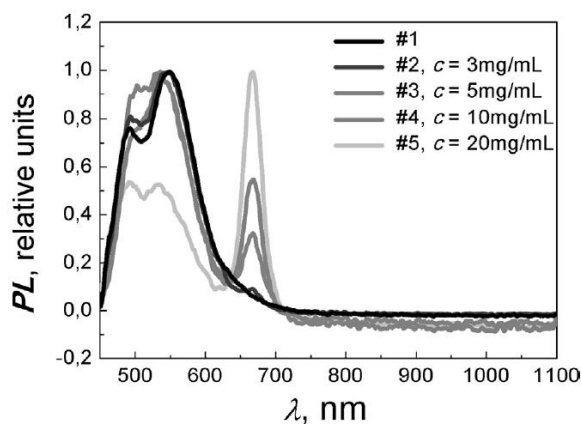


Fig. 54. Normalized *PL* spectra of all devices when they are excited with UV LED (400 nm light at the peak)

In this graph we can see that it is possible to have Förster energy transfer from Ir(Fppy)₃ to CdS_xSe_{1-x}/ZnS QDs. However it should be noted that large emission peaks at 460 nm and at 550 nm are also observed in TPD/Ir(Fppy)₃/QD/Alq₃ *PL* spectra. These results demonstrate that Förster energy transfer between Ir(Fppy)₃ and CdS_xSe_{1-x}/ZnS QDs is incomplete, and this incomplete energy transfer gives broad emission spectra in our devices.

5. Electroluminescence

A further step in the study was an investigation of electroluminescence properties. Traditional hole and electron transport materials TPD (N,N'-Bis(3-methylphenyl)-N,N'-diphenylbenzidine) and Alq₃ (Tris-(8-hydroxyquinoline)aluminium) were chosen for this purpose. In our previous work [5] we used structure ITO/TPD/EmL/Alq₃/LiF/Al to enhance the intensity of OLEDs by improving charge injection into the emissive layer. However, a clear impact of Alq₃ emission was noticed in OLED *EL* spectra. To overcome this problem a hole-blocking layer from TmPyPB material (3,3'-[5'-[3-(3-Pyridinyl)phenyl][1,1':3',1''-terphenyl]-3,3''-diyl]bispyridine) was added (a full structure of OLED devices is sketched in **Figure 68**), which led to positive results – no Alq₃ spectral influence was seen (*EL* spectrum of control device ITO/TPD/TmPyPB/Alq₃/LiF/Al is

shown in **Figure 66**). But such effective blocking caused accumulation of holes not only in EML, but in TPD area, too.

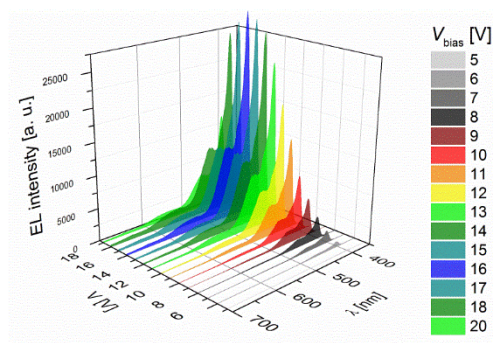


Fig. 66. *EL* spectrum dependence on voltage of the control device

ITO/TPD (40 nm)/TmPyPB (40 nm)/Alq₃ (40 nm)/LiF (1 nm)/Al [6]

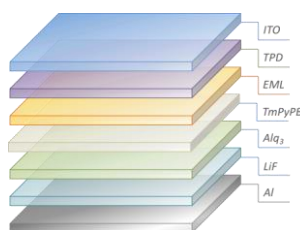


Fig. 68. A scheme of multilayer OLED structure used for investigation of electroluminescence properties. Various newly synthesized compounds were tested as an emissive layer (EmL) [6]

The *EL* spectra of OLEDs using chinoline and 1,8-naphtyridine derivatives in EmL are presented in subsections **5.1.** and **5.2.** respectively.

Also in subsection **5.3.** we are going to present hybrid OLEDs with structure ITO/TPD/Ir(Fppy)₃/QD/Alq₃/LiF/Al where instead hole blocking layer from TmPyPB material the thin layer of CdS_xSe_{1-x}/ZnS QDs is used.

5.1. Electroluminescence properties of OLEDs based on chinoline derivatives

In this section we present the primary study results on electroluminescence of investigated chinoline materials, used as an emissive layer in thermally evaporated OLED structure. OLEDs based on chinoline derivatives (**2a**), (**2b**), (**3a**), (**3b**), (**4a**) and (**5a**) did

not show any electroluminescence due to polycrystal nature of the emissive layers. The *EL* spectra of the other OLEDs are shown in **Figure 69**, **Figure 71**, **Figure 73** and **Figure 75**.

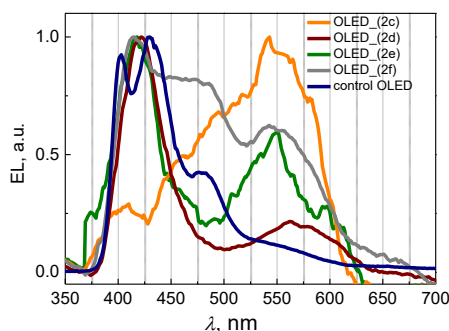


Fig. 69. *EL* spectra of OLEDs based on 2 group chinoline derivatives

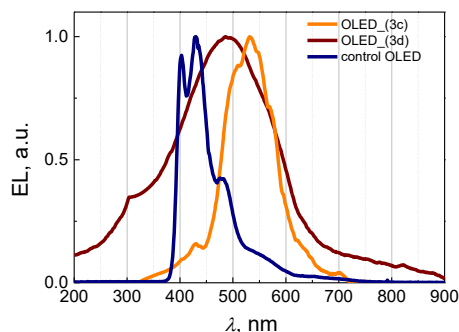


Fig. 71. *EL* spectra of OLEDs based on 3 group chinoline derivatives

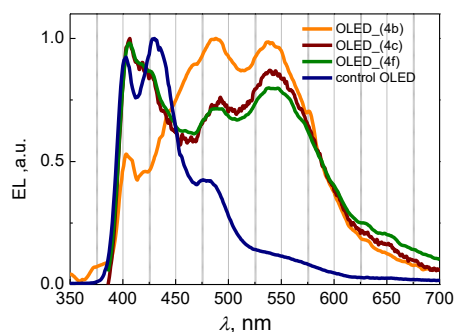


Fig. 73. *EL* spectra of OLEDs based on 4 group chinoline derivatives

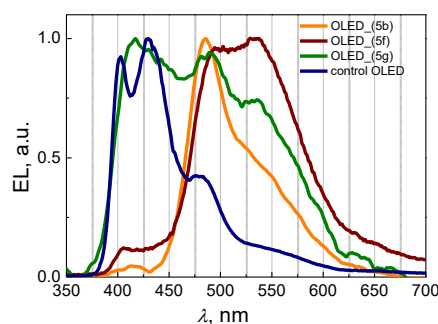


Fig. 75. *EL* spectra of OLEDs based on 5 group chinoline derivatives

Broad *EL* spectra of OLEDs based on quinoline derivatives ((**3d**), (**4b**), (**4c**), (**4f**) and (**5g**)) depend on emissive layer *QY* value – bigger the value, the more emission we get from emissive layer; and TPD electroluminescence. Over time TPD layer tends to crystallize [26] and the spectral components of the whole *EL* are changing. This is big drawback because the life time of the broad spectra OLEDs shortens.

5.2. Electroluminescence properties of OLEDs based on 1,8-naphtyridine derivatives

In this section we present the primary study results on electroluminescence of investigated 1,8-naphtyridine materials, used as an emissive layer in thermally evaporated OLED structure. Roughly summarizing spectral characteristics (*EL* spectra are shown in **Figure 78**, **Figure 80** and **Figure 82**), we observed *EL* covering almost all colour spectra:

- Camphor group emitted lettuce (material **(28)**) and blue-green (material **(29)**) colour light;
- Dioxy group – green (materials: **(31)**, **(34)** and **(36)**);
- Dithio group – yellow (materials: **(33)**, **(35)** and **(37)**);
- Smallest molecules from different groups – orange-red (materials: **(15)**, **(30)** and **(32)**);
- Oxo group – greenish close to white (materials: **(17)**, **(18)**, **(26)** and **(27)**);
- Duplex group – cold and warm white (materials: **(16)**, **(19)**, **(21)**).

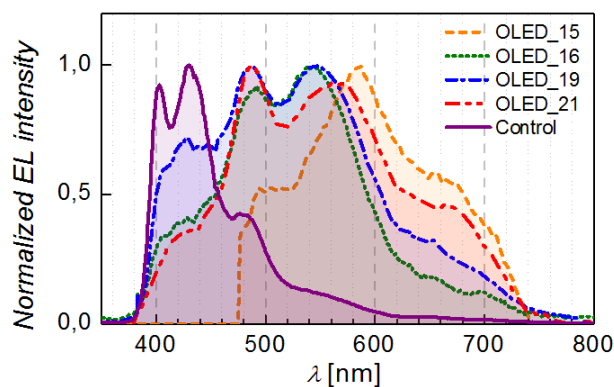


Fig. 78. *EL* spectra of OLEDs with duplex molecules group EmL [6]

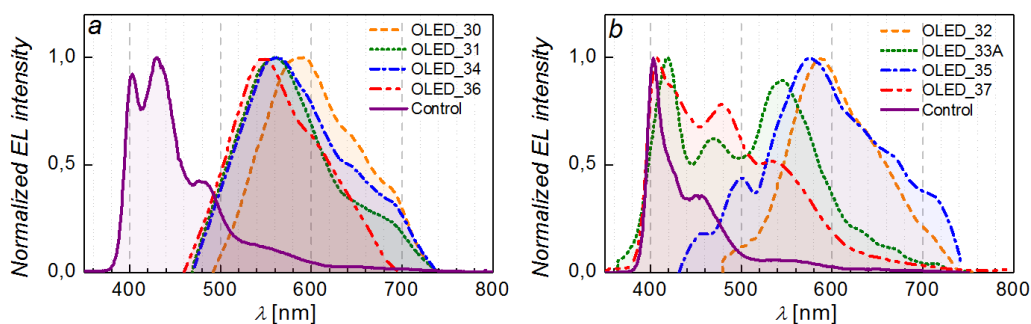


Fig. 80. **a)** *EL* spectra of OLEDs with dioxy group EmL, **b)** *EL* spectra of OLEDs with dithio group EmL

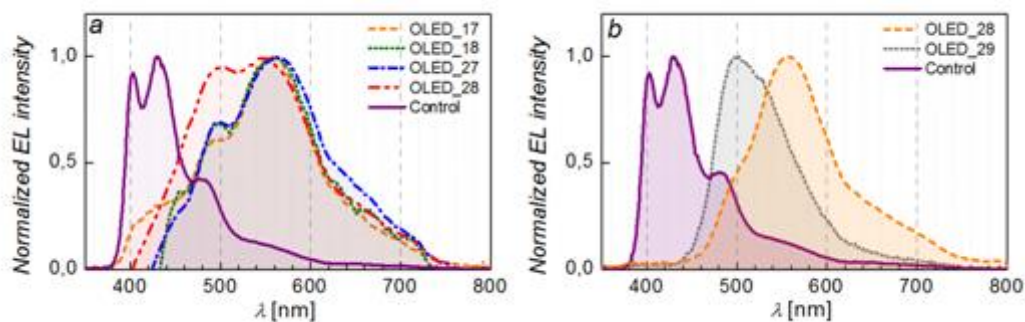


Fig. 82. a) *EL* spectra of OLEDs with oxo group EmL, b) *EL* spectra of OLEDs with camphor group EmL

We observed a broad *EL* spectrum from duplex molecules (**Figure 78**) having a non-planar bicyclic fragment in the middle. *QY* of emissive layers of 1,8-naphthyridine derivatives are far bigger than of chinoline derivatives. These 1,8-naphthyridine based molecules tend to shape themselves in “V” form. This form can allow to appear more energy interstates from TPD/EmL interlayer for radiative recombination [27]: we can observe *EL* in ~ 660 nm range (**Figure 78**) which corresponds the radiative transition from EmL LUMO_{opt} level (taking into account 0,5 eV discrepancy from LUMO level) to TPD HOMO level [6].

All in all, *EL* spectral characteristics of OLEDs not only depend on molecule’s chemical structure, but also its spatial conformation, effective volume and packing properties influenced by the former two. By controlling molecular structure and layer thicknesses one can obtain *EL* emission of desired spectral composition. Wise selection of thicknesses of emissive and supporting layers can also lower turn-on voltage and improve device efficiency, however, this requires cumbersome experimental (and/or modelling) work and was not under the scope of this research stage.

5.3. Electroluminescence properties of QD OLEDs

Previous *PL* results (shown in subsection 4.3.2.) demonstrated that Förster energy transfer between Ir(Fppy)₃ and CdS_xSe_{1-x}/ZnS QDs is incomplete, and this incomplete energy transfer gives broad emission spectra in ITO/TPD/Ir(Fppy)₃/QD/Alq₃/LiF/Al devices (#2, #3, #4) and in ITO/TPD/Ir(Fppy)₃/Alq₃/LiF/Al device (#1) (**Figure 86**).

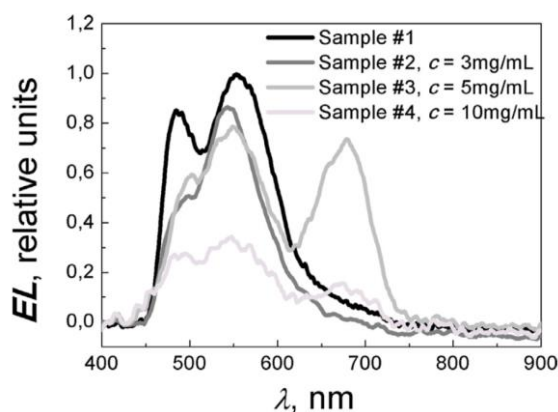


Fig. 86. The *EL* spectra of all devices (#1, #2, #3, #4)

At first we have made control ITO/TPD/Ir(Fppy)₃/Alq₃/LiF/Al device (#1) to know in which spectral region emits Ir(Fppy)₃ material layer. From **Figure 86** (for device #1) we can see that the impact of Alq₃ emission in ~ 535 nm region [5, 28] is small and the electroluminescence spectrum of the control device coincides the *PL* spectrum of single Ir(Fppy)₃ layer. It is clearly seen that we have *EL* from 450 nm to 750 nm range, we also have got small amount of emission in visible red 620–700 nm spectral regions. CdS_xSe_{1-x}/ZnS QDs emit in red visible spectral region with emission maximum at 680 nm wavelength, so adding extra QDs emission layer should increase *EL* in red region in our device.

In **Figure 86** we can see *EL* spectra of devices with different concentrations of QDs: 3 mg/ml, 5 mg/ml and 10 mg/ml. Though we could see in *PL* spectra (**Figure 54**) intense emission of QDs with concentration of QDs 20 mg/mL, this device did not show any *EL*. The layer of QDs is too thick so both charges the electrons and the holes were trapped in different QDs and could not recombine by emitting photons. In the case, when concentration of QDs in toluene is small for e.g. 3 mg/mL we could not observe any emission from QDs (**Figure 86**), because QDs have not formed uniform layer and there is no complete coverage of the Ir (Fppy)₃ with QDs. When concentration of QDs is 10 mg/mL it is clearly seen in *PL* spectrum that excitation in Ir(Fppy)₃ is stronger compared to excitation in QDs. The same tendency is observed in *EL* spectrum in the same device. By comparing samples with 5 mg/ ml, 10 mg/ml and 20 mg/ml concentrations of QDs it could be noticed that excitation of Ir(Fppy)₃ in *PL* spectra is becoming weaker and *PL* of

QDs is getting more intense while increasing concentration. But we can see from **Figure 86** that *EL* spectra of these samples with $c = 5$ mg/mL, 10 mg/mL differs from *PL* spectra: with smaller concentration of QDs we are getting more light from QDs in *EL* spectra. Therefore, it could be concluded that the *EL* peak at 678 nm in sample with $c = 5$ mg/mL appears because there are two QD excitation mechanisms: charge injection and energy transfer from Ir(Fppy)₃.

In conclusion, we have fabricated broad spectra light-emitting diodes based on two emission layers: organic material Ir(Fppy)₃ and CdS_xSe_{1-x}/ZnS QDs with different concentrations of QDs. The emission spectrum of Ir(Fppy)₃ and the simultaneous existence of incomplete energy transfer from Ir(Fppy)₃ to CdS_xSe_{1-x}/ZnS QDs and carrier trapping in CdS_xSe_{1-x}/ZnS QDs leads to broad light emission in visible spectrum. This is the simplest component and structure in the realization of broad spectrum hybrid QD OLEDs. Our results show that an organic and inorganic hybrid with proper concentration as the active medium could be a promising route to broad spectra QD OLEDs.

6. Main results and conclusions

In this dissertation we have presented benzo- and naphthoquinoline derivatives (based on already known synthons) with different substituents: chlorine, tetrazole, iminophosphorane, amine; and 1,8-naphthyridine based compounds having a non-planar bicyclic moiety: bicyclo[3.3.1]nonane, bicyclo[3.3.0]octane and camphor which were synthesized at the Faculty of Chemistry, Vilnius university. Also we have presented these materials as potential emitters in multi-layered OLED systems. In addition, to improve broad spectra stability the hybrid OLEDs (QD OLEDs) with Ir(Fppy)₃ and CdS_xSe_{1-x}/ZnS quantum dots (QDs) as emissive layers (EmL) were shown too.

The main findings are as follows:

- The value of E_{g_opt} energy increases in all quinoline groups while substituents are changed as follows: amine, tetrazole, iminophosphorane and chlorine. Additional benzene rings in the molecules lowered E_{g_opt} energy value and shifted *PL* and absorbance spectra peaks towards shorter wavelengths.
- OLEDs need emissive layers with high *QY* values. The layer of (**5b**) quinoline derivative had biggest *QY* reaching 10%. The layers of quinoline derivatives with tetrazole

substituents luminescated poorly except **(3c)** material (thin layer $QY = 5\%$). The QY s of chinoline derivatives with amine substituents were 4% - 10%. While the luminescence properties of chinoline derivatives with chlorine and iminophosphorane side groups were the worst.

- OLEDs with structure ITO/TPD/EmL/TmPyPB/Alq₃/LiF/Al showed EL spectra in blue and green region when **(2c)**, **(2d)**, **(2e)**, **(2f)** and **(3c)**, **(5b)**, **(5f)** chinoline derivatives were used in the emissive layer. When **(3d)**, **(4b)**, **(4c)**, **(4f)** and **(5g)** derivatives were used as EmL in ITO/TPD/EmL/TmPyPB/Alq₃/LiF/Al OLEDs the broad visible spectra light were obtained due to EL components originated from emissive layer and TPD material. Over time TPD layer tends to crystallize and the spectral components of the whole EL as well as CRI values are changing. This is big drawback because the life time of the broad spectra OLEDs shortens.

- Wide variation of molecular structure let us obtain wide-ranging $E_{HOMO} = [-5.9; -5.4]$ eV, $E_{LUMO} = [-3.4; -2.4]$ eV and $E_{g,opt} = [2.3; 3]$ eV values, that can be adjusted for one's needs in respect of energetic compatibility among material layers in a device. The bathochromic shift between PL spectra peaks in the solutions and in the layers of the same materials was observed. **(18)**, **(36)** and **(37)** 1,8-naphtyridine derivatives had the longest π conjugation in the group and PL QY in the layer of these materials reached 16%, 4% and 11% respectively.

- OLEDs with structure ITO/TPD/EmL/TmPyPB/Alq₃/LiF/Al showed EL spectra from red-orange to blue region when 1,8-naphtyridine derivatives: **(15)**, **(32)**, **(35)**, **(30)**, **(28)**, **(34)**, **(31)**, **(36)**, **(17)**, **(18)**, **(26)**, **(27)**, **(33)** and **(29)** were used in the EmL.

- OLEDs based on "V" shaped duplex molecules: **(16)**, **(19)** and **(21)**; with non-planar bicyclo[3.3.1]nonane moiety in the centre emitted broad white EL spectrum due to emission of emissive layer as well as emission of TPD/EmL interlayer.

- Chinoline, 1,8-naphtyridine derivatives and CdS_xSe_{1-x}/ZnS QDs in toluene were not chemically compatible because toluene dissolved chinoline and 1,8-naphtyridine derivatives. For that reason hybrid OLEDs were made using Ir(Fppy)₃ material which was chemically compatible with the CdS_xSe_{1-x}/ZnS QDs.

- The optimal concentration of QDs in toluene is 5mg/ml for hybrid OLED. The wide EL emission properties of Ir(Fppy)₃ and the simultaneous existence of

incomplete energy transfer from Ir(Fppy)₃ to CdS_xSe_{1-x}/ZnS QDs and carrier trapping directly on CdS_xSe_{1-x}/ZnS QDs led to broad light emission in visible spectrum. This is the simplest component and structure in the realization of broad spectrum QD OLEDs.

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List of publications related to the thesis:

1. S. Višniakova, I. Urbanavičiūtė, L. Daukšaitė, M. Janulevičius, B. Lenkevičiūtė, I. Sychugov, K. Arlauskas, A. Žilinskas. Luminescent benzo- and naphthoquinolines: Synthesis and investigation of photophysical properties. *J. Lumin.*, 2015. Vol. 167, p. 261.
2. I. Urbanavičiūtė, S. Višniakova, J. Dirsytė, G. Juška, B. Lenkevičiūtė, E. Bužavaitė, A. Žilinskas, K. Arlauskas. A series of new luminescent non-planar 1,8-naphthyridine derivatives giving coloured and close-to-white electroluminescence spectra. *J Lumin.*, 2017. Vol. 181, p. 299.
3. B. Lenkeviciute, M. Vitkus, G. Juska, K. Genevicius. Hybrid OLEDs with CdS_xSe_{1-x}/ZnS core-shell quantum dots: An investigation of electroluminescence properties. *Synth. Met.*, 2015. Vol. 209, p. 343

List of presentations at the conferences (underlined Bronė Lenkevičiūtė – presented personally):

1. Bronė Lenkevičiūtė, Kęstutis Arlauskas, Investigation of Photoelectric Features of Vacuum Deposited Organic Materials Ir(Fppy)₃ and Alq₃ Layers, European Material Research Society conference, Srastbourg, 2013.
2. Bronė Lenkevičiūtė, Kęstutis Arlauskas, Investigation of Photoelectric Features of Vacuum Deposited Organic Materials Ir(Fppy)₃ and Alq₃ Layers, 15th International Conference Advanced Materials and Technologies, Palanga, 2013.
3. Bronė Lenkevičiūtė, Kęstutis Arlauskas, Investigation of Photoelectric Features of Vacuum Deposited Organic Materials Ir(Fppy)₃ and Alq₃ Layers, International Winterschool on Bioelectronics BioEl2014, Tirol, 2014.
4. Bronė Lenkevičiūtė, Mantas Vitkus, Kęstutis Arlauskas, Kristijonas Genevicius, Giedrius Juška, Investigation of Electroluminescence Features of Vacuum Deposited Organic Materials TPD, Ir(Fppy)₃, Alq₃ and Spin-coated CdS_xSe_{1-x}/ZnS Quantum Dots Layers, Nanotechnology: Research and Development 2014 conference, Vilnius, 2014.

5. Bronė Lenkevičiūtė, Mantas Vitkus, Kęstutis Arlauskas, Kristijonas Genevičius, Giedrius Juška, Investigation of Electroluminescence Features of Vacuum Deposited Organic Materials TPD, Ir(Fppy)₃, Alq₃ and Spin-coated CdS_xSe_{1-x}/ZnS Quantum Dots Layers, 16th International Conference Advanced Materials and Technologies, Palanga, 2014.
6. J. Dirsytė, S. Višniakova, I. Urbanavičiūtė, B. Lenkevičiūtė, K. Arlauskas, A. Žilinskas, Synthesis and Photophysical Investigation of Different 2-Aminoquinoline-3-Carbaldehydes, International conference Chemistry and chemical technology 2014, Kaunas, 2014.
7. J. Dirsytė, S. Višniakova, I. Urbanavičiūtė, B. Lenkevičiūtė, K. Arlauskas, A. Žilinskas, Synthesis of 1,8-Naphthyridine Compounds Fused With Bicyclic Moieties, International conference Balticum Organicum Syntheticum 2014, Vilnius, 2014.
8. I. Urbanavičiūtė, B. Lenkevičiūtė, J. Dirsytė, S. Višniakova, K. Arlauskas, A. Žilinskas, Luminescent 2-aminoquinoline-3-carbaldehydes: synthesis and investigation of their electric, photoelectric and optical properties, HOMERIC Fall School 2014, Bordo, 2014.
9. Brone Lenkeviciute, Mantas Vitkus, Kestutis Arlauskas, Giedrius Juska, Investigation of Electroluminescence Features of Vacuum Deposited Organic Materials TPD, Ir(Fppy)₃, Alq₃ and Spin-coated CdS_xSe_{1-x}/ZnS Quantum Dots Layers, HOMERIC Fall School 2014, Bordo, 2014.
10. Brone Lenkeviciute, Mantas Vitkus, Giedrius Juska, Kestutis Arlauskas, Investigation of Electroluminescence Features of Vacuum Deposited Organic Materials TPD, Ir(Fppy)₃, Alq₃ and Spin-coated CdS_xSe_{1-x}/ZnS Quantum Dots Layers, Lithuanian New Year Conference of Physics, Vilnius, 2015.
11. Brone Lenkeviciute-Vasiliauskiene, Indre Urbanaviciute, Sigita Visniakova, Justina Dirsyte, Giedrius Juska, Ernesta Buzavaite, Albinas Zilinskas, Kestutis Arlauskas, A Series of New Luminescent Non-Planar 1,8-naphthyridine Derivatives Giving Coloured and Close-to-White Electroluminescence Spectra, International Conference on Synthetic Metals 2016, Guangzhou, 2016.