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«ҚАЗАҚСТАН РЕСПУБЛИКАСЫ  
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫ» РҚБ

# Х А Б А Р Л А Р Ы

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## ИЗВЕСТИЯ

РОО «НАЦИОНАЛЬНОЙ  
АКАДЕМИИ НАУК РЕСПУБЛИКИ  
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## **INCLUSION OF AZULENE STRUCTURAL UNITS IN THE BASIS OF CONJUGATED POLYMERS: IMPROVEMENT OF PROTON SENSITIVITY AND FLUORESCENCE**

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**Abstract.** The growing interest in aromatic polymer compounds with an extended  $\pi$ -electron coupling system is explained by their importance as functional materials for organic optoelectronics. At present, much attention is paid to arylated, as well as substituted by electron acceptor and/or electron donor groups, aromatic and heteroaromatic compounds. One of the advantages of such conjugated aromatic systems is that they can fine-tune the electronic structure of materials in order to optimize their performance and morphology. In this regard, the structural isomer of naphthalene - azulene is of considerable interest. The nonalternant aromatic hydrocarbon azulene has

unique electronic and spectral properties, including a polarized structure with a dipole moment of the order of 1,08 D and abnormal Anti-Kasha fluorescence  $S_2 \rightarrow S_0$ .

The article discusses the synthesis of two fluorene-azulene  $\pi$ -conjugated copolymers using the modern methodology of organic synthesis as the Suzuki – Miyaura -cross-coupling reaction.

It was shown that the interaction of 1,3-dibromazulene, as well as 2,7-Bis(3-bromoazulen-1-yl)-9,9-dioctyl-fluorene with 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl) bis(1,3,2-dioxaborinane) under Suzuki – Miyaura cross-coupling conditions, azulene conjugate copolymers were synthesized: poly[2,7-(9,9-dioctylfluorenyl)-alt-(1',3' - azulenyl)] and poly[1,3-bis(9',9'-dioctylfluoren-2'-yl)azulenyl]-alt-[1'',3'' - azulenyl]. It is revealed that neutral solutions of the obtained copolymers are nonfluorescent, but they become luminescent upon addition of trifluoroacetic acid. The «launching» of fluorescence occurs as a result of the formation of a 6  $\pi$ -electron azulenyl cation, which rearranges the general electronic character of polymers, in particular, HOMO-LUMO levels and band gap.

The obtained results demonstrate that the introduction of azulene units into a conjugated polymer framework has great potential for application as new protic functional materials.

**Key words:** azulene, azulene copolymers, cross- coupling, photophysical properties.

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## **ҚОСАРЛАНҒАН ПОЛИМЕРЛЕРДІҢ НЕГІЗІНЕ АЗУЛЕНДІК ҚҰРЫЛЫМДЫҚ БІРЛІКТЕРДІ ҚОСУ: ПРОТОНҒА СЕЗІМТАЛДЫҚ ПЕН ФЛУОРЕСЦЕНЦИЯНЫ ЖАҚСARTU**

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**Аннотация:** Кеңейтілген  $\pi$ -электронды қосарланған жүйесі бар ароматты полимерлі қосылыстарға қызығушылықтың артуы олардың органикалық оптоэлектроника үшін функционалды материалдар ретіндегі маңыздылығына байланысты. Қазіргі уақытта арилденген, сондай-ақ электрондонорлы және/немесе электронакцепторлы топтармен алмастырылатын ароматты және гетероароматты қосылыстарға көп көңіл бөлінеді. Осындай ароматты жүйелердің артықшылықтарының бірі - материалдардың өнімділігі мен морфологиясын оңтайландыру мақсатында олардың электрондық құрылымын дәл реттей алады. Осыған байланысты нафталиннің азулен атты құрылымдық изомері үлкен қызығушылық тудырады. Альтернанты емес ароматты көмірсутек азуленінің бірегей электронды және спектрлік қасиеттері бар, оның ішінде дипольдік моменті шамамен 1,08 D болатын поляризацияланған құрылым және аномальды  $S_2 \rightarrow S_0$  анти-Каш флуоресценциясы.

Жұмыста Сузуки-Мияура кросс-комбинация реакциясы ретінде заманауи органикалық синтез әдістемесін пайдалана отырып, екі фтор-азулен  $\pi$ -конъюгацияланған сополимерлердің синтезі талқыланады. Сузуки-Мияура кросс-бірігу реакциясының жағдайында 1,3-дибромазулен, сондай-ақ 2,7-бис(3-бромазулен-1-ил)-9,9-диоктил-флуорен 2,2'-(9,9-диоктил-9Н-флуорен-2,7-диил)бис(1,3,2-диоксиборинанмен) әрекеттескенде келесі азулен қосарланған сополимерлер синтезделді: поли[2,7-(9,9-диоктилфлуоренил)-альт-(1',3' - азуленил)] және поли[1,3-бис(9',9'-диоктилфлуорен-2'-ил)азуленил]-альт-[1'',3'' - азуленил]. Алынған сополимерлердің бейтарап ерітінділері флуоресцентті емес, бірақ олар трифторсірке қышқылын қосқанда люминесцентті болады. Флуоресценцияның "іске қосылуы" полимерлердің жалпы электрондық сипатын, атап айтқанда НОМО-LUMO деңгейлерін және жолақ зоны өзгертетін 6  $\pi$  электроны бар азуленил катионының түзілуінен туындайды.

Нәтижелер қосарланған полимер қаңқасына азулен бірліктерін енгізу жаңа протонды функционалды материалдар ретінде қолдану үшін үлкен әлеуетке ие екенін көрсетеді.

**Түйін сөздер:** азулен, азулен сополимерлері, кросс-комбинация, фотофизикалық қасиеттер.

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## **ВКЛЮЧЕНИЕ АЗУЛЕНОВЫХ СТРУКТУРНЫХ ЕДИНИЦ В ОСНОВУ СОПРЯЖЕННЫХ ПОЛИМЕРОВ: УЛУЧШЕНИЕ ПРОТОННОЙ ЧУВСТВИТЕЛЬНОСТИ И ФЛУОРЕСЦЕНЦИИ**

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**Аннотация.** Растущий интерес к ароматическим полимерным соединениям с расширенной системой  $\pi$ -электронного сопряжения объясняется их важностью в качестве функциональных материалов для органической оптоэлектроники. В настоящее время большое внимание уделяется арилированным, а также замещенным электроноакцепторными и/или электронодонорными группами ароматическим и гетероароматическим соединениям. Одним из преимуществ таких сопряженных ароматических систем является то, что они могут точно настроить электронную структуру материалов с целью оптимизации их производительности и морфологии. В этом плане значительный интерес представляет структурный изомер нафталина азулен. Неальтернативный ароматический углеводород азулен обладает уникальными электронными и спектральными свойствами, в том числе поляризованной структурой с дипольным моментом порядка  $1,08 \text{ D}$  и аномальной анти-Каша флуоресценцией  $S_2 \rightarrow S_0$ .

В работе обсуждается синтез двух флуорен-азуленовых  $\pi$ -сопряженных сополимеров, посредством современной методологии органического синтеза как реакции кросс-сочетания Сузуки–Мияуры. Показано, что взаимодействием



1,3-дибромазулена, а также 2,7-Bis(3-bromoazulen-1-yl)-9,9-dioctyl-fluorene с 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl)bis(1,3,2-dioxaborinane) в условиях сочетания Сузуки–Мияуры были синтезированы азуленовые сопряженные сополимеры - poly[2,7-(9,9-dioctylfluorenyl)-alt-(1',3' - azuleny)] и poly[1,3-bis(9',9'-dioctylfluoren-2'-yl)azulenyl]-alt-[1'',3'' - azuleny]. Выявлено, что нейтральные растворы полученных азуленовых сополимеров не являются флуоресцентными, но они становятся светящимися при добавлении трифторуксусной кислоты. Показано, что «включение» флуоресценции происходит за счет образования  $\pi$ -электронного азуленил-катиона, который перестраивает общий электронный характер полимеров, в частности, уровни фронтальных орбиталей НОМО – LUMO и ширину запрещенной зоны.

Полученные результаты демонстрируют, что введение азуленовых звеньев в сопряженный полимерный каркас имеют большой потенциал для применения в качестве новых протонных функциональных материалов.

**Ключевые слова:** азулен, сополимеры азулена, кросс-сочетание, фотофизические свойства

## Introduction

Azulene and its compounds as nonalternant aromatic substances is the subjects of much scientific research (Lash, et al, 2007, Wakabayashi, et al, 2007; López-Allied, et al, 2021; Xin, et al, 2021). This interest was due to its unusual electronic structure and abnormal photophysical properties, including a polarized structure with a dipole of the order of 1.08 D (Anderson, et al, 1959) and unusual anti-Kasha's fluorescence  $S_2 \rightarrow S_0$  (Tomin, et al, 2018; Nenov, et al, 2018), Dunlop, 2023).

The potential of azulene makes it the object of widespread research in the field of functional materials, such as conductive polymers, optoelectronic molecular switches, field-effect transistors (OFETs), light-emitting diodes (OLED), solar cells (OSCs), nonlinear optical (NLO) and other materials (Wang, et al, 2003; Wang, et al, 2004; Mrozek, et al, 2001; Dong, et al, 2016; Ou, et al, 2019).

The literature describes the synthesis of azulene-containing polymers. For example, the obtained polyazulene was first reported in the article (Kilara, et al, 1997) Later, (Wang, et al, 2003; Wang, et al, 2003) showed new methods for producing polyazulene, as well as the synthesis of a number of highly conductive azulene-containing polymers.

The structure of azulene **1** (Figure 1) can be considered as a tropylium cation condensed with a cyclopentadienyl anion. The electron-saturated cyclopentane azulene ring can rapidly undergo electrophilic substitution at  $C_1$  or  $C_3$  to form a stable aromatic azulene cation with 6  $\pi$ -electrons. The formation of such cation in a number of azulene compounds has been reported to lead to interesting electrochemical and electrochromic properties (Sprutta, et al, 2007; Ito, 2005). Therefore, such simple chemical conversion of azulene units resulting in the formation of cations in azulene-containing polymers can be used to adjust the overall physical properties of the polymers, such as "launching" fluorescence or change in electrical conductivity, etc.

Thus,  $\pi$ -conjugated azulene-based polymers are expected to readily undergo an acid-

base or redox reaction to form azulenyli cations/radical cations and potentially function as efficient optoelectronic materials.

In this work we synthesize two azulene  $\pi$ -conjugated copolymers poly[2,7-(9,9-dioctylfluorenyl)-alt-(1',3' - azulenyli)] **3** and poly[1,3-bis(9',9'-dioctylfluorene-2'-yl) azulenyli]-alt-[1'',3'' - azulenyli] **5**, by Suzuki – Miyaura cross-coupling reaction using Pd<sub>2</sub>(dba)<sub>3</sub> as catalyst. Such molecular construct exploits both the dipole moment of the azulene unit and its ability to acquire and lose protons in the electron-donor five-membered cycle.

### Methods and Materials

<sup>1</sup>H and <sup>13</sup>C NMR spectra were taken on a Jeol ECA-500 spectrometer [operating frequency 500.15 MHz (<sup>1</sup>H) and 100.61 MHz (<sup>13</sup>C)] in CDCl<sub>3</sub>, internal standard – TMS. IR spectra were recorded on a Fourier spectrometer Avatar-360 in KBr tablets. Mass spectra were obtained on a Thermo Scientific LTQ FT Ultra spectrometer. Elemental analysis was performed on the instrument EuroVektorEA-3000. Absorption spectra were taken on a Shimadzu UV-1800 spectrophotometer. Fluorescence spectra were recorded on an Agilent Cary Eclipse spectrofluorimeter. Thermogravimetric studies were performed on a TGA Q500 instrument (in current N<sub>2</sub>; heating at 10 ° per minute; interval 20-500°C). Differential scanning calorimetry was performed on a DSC Q2000 (in current N; heating at 5 ° per minute; interval 20-300 °C). Gel permeation chromatography was performed on Agilent PL-GPC 220 (polystyrene standard; THF eluent). The melting points were determined on a Melting Point M-560 apparatus.

Commercially available reagents and solvents: azulene, 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl)bis(1,3,2-dioxaborinane), Pd<sub>2</sub>(dba)<sub>3</sub>, Pd(PPh<sub>3</sub>)<sub>4</sub>, P(o-tol)<sub>3</sub>, Et<sub>4</sub>NOH, Ag<sub>2</sub>CO<sub>3</sub>, TFA, THF and others were used without further purification. Dibromoazulene **2** was synthesized according to the literary method (Anderson, et al, 1953).

### Synthesis and description of copolymers

*Poly[2,7-(9,9-dioctylfluorenyl)-alt-(1',3' - azulenyli)] 3.* To a solution of 177 mg (0.62 mmol) of compound **2** and 345 mg (0.62 mmol) of 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl)bis(1,3,2-dioxaborinane) in 50 mL toluene under argon were added 17 mg (0.02 mmol) of Pd<sub>2</sub>(dba)<sub>3</sub>, 13 mg (0.04 mmol) of P(o-tol)<sub>3</sub> and 2 mL of tetraethylammonium hydroxide. The reaction mixture was stirred for 20 hours at 80 °C. After completion of the reaction, 100 ml of chloroform was added to the reaction flask, followed by washing with water in a separatory funnel. The resulting chloroform phase was concentrated in vacuo to 10 mL and added to 300 mL of methyl alcohol. The resulting precipitate was purified by various organic solvents in a Soxhlet apparatus. The product was then recovered by chloroform, precipitated from methyl alcohol, dried under vacuum. A dark orange solid was obtained. Yield 230 mg (65%). Anal. Calcd. For (C<sub>39</sub>H<sub>46</sub>)<sub>n</sub>: C 90.95; H 9.05; Found: C 89.92; H 8.65. Mn = 19.5 kDa; PDI = 2.15. <sup>1</sup>H NMR:  $\delta$  8.55-8.60 (m, 2H), 8.23-8.27 (m, 1H), 7.61-7.85 (m, 7H), 7.10-7.14 (m, 2H), 2.06 (br, m, 4H), 0.75-1.13 (m, 30H). <sup>13</sup>C NMR:  $\delta$  151.52, 140.10, 139.63, 137.31, 137.64, 136.52, 136.51, 131.62, 129.41, 124.72, 123.64, 120.53, 55.52, 40.76, 32.54, 30.52, 29.63, 29.75, 24.55, 23.13, 14.24.

*2,7-Bis(3-bromoazulen-1-yl)-9,9-dioctyl-fluorene 4.* To the solution of 200 mg (0.70

mmol) of compound **2** and 155 mg (0.28 mmol) of 0,28 ммоль 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl)bis(1,3,2-dioxaborinane) in 8 ml of tetrahydrofuran under argon were added 23 mg (0.02 mmol) of Pd(PPh<sub>3</sub>)<sub>4</sub> and 220 mg (0.8 mmol) of silver (I) carbonate. The reaction mixture was stirred for 16 hours at 80 °C. After completion of the reaction, 100 ml of water was added, followed by extraction with methylene chloride (3 × 50 ml). The organic phase was dried over MgSO<sub>4</sub>, and the solvent was distilled off in vacuo. The residue was purified by silica gel column chromatography (hexane-ethyl acetate, 9:1). A dark brown solid was obtained. Yield 480 mg (86%). HRMS (EI): m/z [M]<sup>+</sup> calcd for C<sub>16</sub>H<sub>12</sub> 800.2515, found 800.2510. <sup>1</sup>H NMR: δ, 8.35 (s, 2H), 8.35 (d, J = 10.0 Hz, 4H), 7.86 (d, J = 7.5 Hz, 2H), 7.82 (s, 2H), 7.70–7.61 (m, 2H), 7.56 (d, J = 10.0 Hz, 4H), 7.35 (s, 4H), 2.11 (d, J = 7.2 Hz, 4H), 1.11 (s, 24H), 0.81 (t, J = 6.8 Hz, 6H). <sup>13</sup>C NMR: δ 152.03, 151.55, 144.32, 140.67, 138.71, 134.55, 127.88, 127.25, 125.26, 123.13, 120.42, 119.22, 55.67, 40.34, 31.85, 30.01, 29.25, 23.92, 22.71, 14.13. IR (ν, cm<sup>-1</sup>): 2928, 2857, 1720, 1574, 1540, 1462, 1393, 1247.

*Poly[1,3-bis(9',9'-dioctylfluorene-2'-yl)azulenyl]-alt-[1'',3'' - azulenyl]* **5**. To the solution of 500 mg (0.62 mmol) of compound **4** and 345 mg (0.62 mmol) of 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl)bis(1,3,2-dioxaborinane) in 50 ml of toluene under argon was added 17 mg (0.02 mmol) of Pd<sub>2</sub>(dba)<sub>3</sub>, 13 mg (0.04 mmol) of P(o-tol)<sub>3</sub> and 2 ml of tetraethylammonium hydrogen. The reaction mixture was stirred for 13 hours at 80 °C. After completion of the reaction, 100 ml of chloroform was added to the reaction flask, followed by washing with water in separatory funnel. The received chloroformic phase was condensed in vacuum to 10 ml and added to 300 ml of methyl alcohol. The resulting precipitate was purified by various organic solvents in a Soxhlet apparatus. The product was then recovered by chloroform, precipitated from methyl alcohol, dried under vacuum. A dark orange solid was obtained. Yield 200 mg (60%). Anal. Calcd. For (C<sub>78</sub>H<sub>92</sub>)<sub>n</sub>: C 90.98; H 9.02; Found: C 89.88; H 8.78. Mn = 58.5 kDa; PDI = 1.76. <sup>1</sup>H NMR: δ 8.22–8.39 (m, 2H), 7.85–7.97 (m, 2H), 7.74–7.85 (m, 2H), 7.66–7.74 (m, 2H), 7.55–7.61 (m, 2H), 7.36–7.50 (m, 2H), 2.05 (m, 4H), 1.05 (m, 24H), 0.75–0.79 (m, 6H). <sup>13</sup>C NMR: δ 151.55, 140.12, 139.63, 137.34, 137.67, 136.53, 131.62, 129.41, 124.73, 123.64, 120.57, 55.56, 40.72, 32.54, 30.56, 29.67, 29.72, 24.54, 23.11, 14.25.

## Results and discussion

Synthesis route leading to azulene conjugated copolymers **3** and **5** are presented in Scheme 1 and Scheme 2. As can be seen from Scheme 1, the key compound **2** was synthesized based on the literature methodology (Anderson, et al, 1953). Further, by cross-coupling Suzuki-Miyaura between dibromide **2** and 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl)bis(1,3,2-dioxaborinane) in toluene in the presence of catalyst Pd<sub>2</sub>(dba)<sub>3</sub>, copolymer **3** was obtained in a good 65% yield.

Another key molecule bis(3-bromoazulenyl)-9,9-dioctylfluorene **4** (Scheme 2) was prepared in 86% yield by combining Suzuki dibromoazulene **2** with 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl)bis(1,3,2-dioxaborinane) in THF in the presence Pd(PPh<sub>3</sub>)<sub>4</sub> as a catalyst. Similarly, the Suzuki-Miyaura reaction between **4** and 2,2'-(9,9-dioctyl-9H-fluorene-2,7-diyl)bis(1,3,2-dioxaborinane) results in polymer **5** with the high 60% yield.

The obtained azulene copolymers **3** and **5** were easily dissolved in organic solvents such as dichloromethane, chloroform, toluene, tetrahydrofuran. The number average molecular weights of the obtained copolymers were determined by GPC (THF eluent). Thus, the  $M_n$  values of polymers **3** and **5** were 19.5 and 58.5 kDa, respectively, and the PDI were 2.15 and 1.76, respectively.

The chemical structure and purity of copolymers **3** and **5** have been proven by NMR and mass spectrometry as well as gel permeation chromatography and elemental analysis.

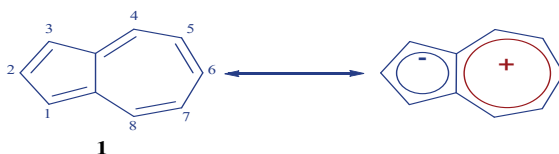
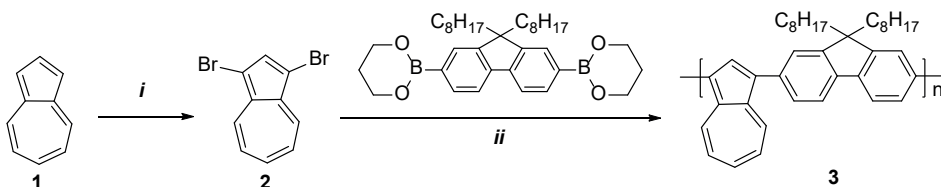
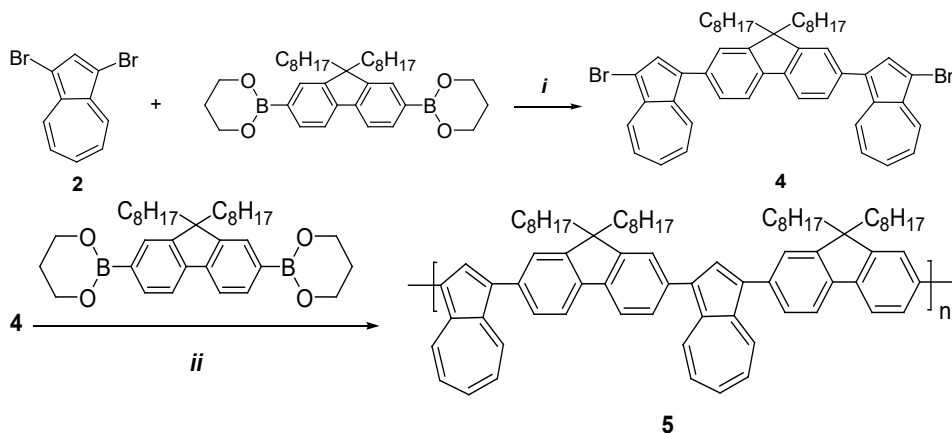


Figure 1. Polarized resonant structure of azulene **1**.



Scheme 1. Synthesis of copolymer **3**: (i) NBS, benzene, room temperature; (ii)  $\text{Pd}_2(\text{dba})_3$ ,  $\text{P}(\text{o-tol})_3$ ,  $\text{Et}_4\text{NOH}$ , toluene,  $85^\circ\text{C}$ .



Scheme 2. Synthesis of copolymer **5**: (i)  $\text{Pd}(\text{PPh}_3)_4$ ,  $\text{Ag}_2\text{CO}_3$ , THF,  $75^\circ\text{C}$ ; (ii)  $\text{Pd}_2(\text{dba})_3$ ,  $\text{P}(\text{o-tol})_3$ ,  $\text{Et}_4\text{NOH}$ , toluene,  $85^\circ\text{C}$ .

The thermal stability of copolymers **3** and **5** was investigated by nitrogen flow thermogravimetric analysis at a heating rate of  $10^\circ\text{C}$  per minute. The onset of degradation **3** and **5** (loss was 5%) was recorded at  $416$  and  $430^\circ\text{C}$ ,

respectively, showing good thermal stability of the synthesized copolymers (Figure 2). Differential scanning calorimetry measurements for copolymers **3** and **5** were performed at the scanning rate of 10 °C per minute. No endothermic or exothermic transition was observed over the entire scanning range of 20 to 300 °C (Figure 3).

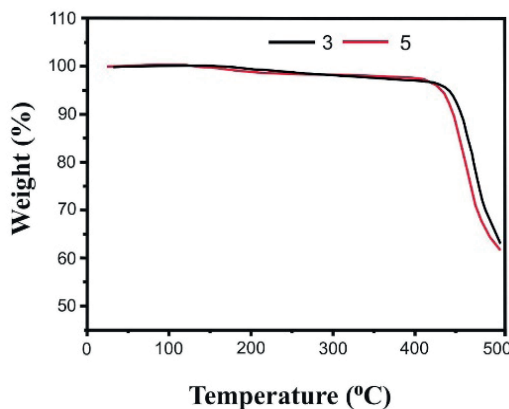


Figure 2. Thermogravimetric measurements of copolymers **3** and **5**.

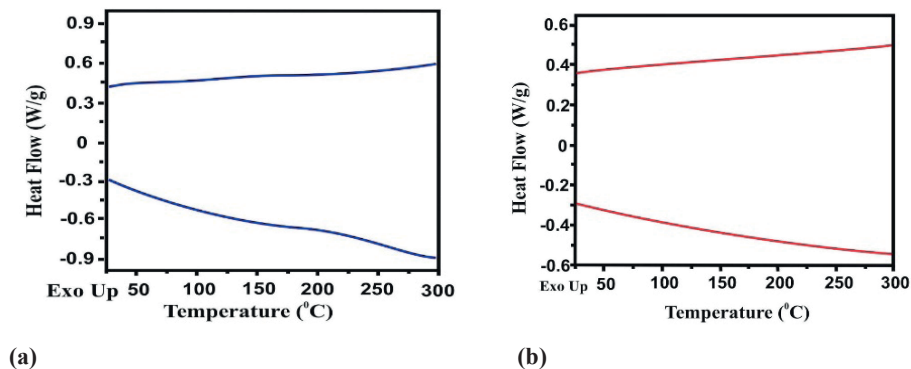


Figure 3. Differential scanning calorimetry measurements of copolymers **3** (a) and **5** (b)

Absorption spectra of copolymers **3** and **5** in the UV-Vis range, including protonated states, are presented in Figure 2, and the corresponding data are summarized in Table 1. In the electron absorption spectra of polymer **3** in tetrahydrofuran (THF), three absorption bands were observed with maxima at 345 ( $\epsilon = 75850$ ) nm, 468 ( $\epsilon = 37120$ ) nm and 675 ( $\epsilon = 1050$ ) nm (Table 1). Similarly, polymer **5** showed two absorption bands  $\pi$ - $\pi^*$  electron junction at 344 ( $\epsilon = 72500$ ) nm and 462 ( $\epsilon = 34900$ ) nm, as well as absorption in the long-wave region at 673 ( $\epsilon = 1015$ ) nm. Weak absorption bands with maxima at 675 (for **3**) nm and 673 (for **5**) nm correspond to the energy transition  $S_0 \rightarrow S_1$ .

Table 1 - Optical data of copolymers **3** and **5**

Copoly- mers	Absorption					Fluorescence <sup>a</sup>	
	<i>Unprotonated</i>			<i>Protonated</i>		<i>Protonated</i>	
	Solvent	$\lambda_{\text{abs}}$ , nm	$\epsilon$ , M <sup>-1</sup> cm <sup>-1</sup>	Solvent <sup>b</sup>	$\lambda_{\text{abs}}$ , nm	Solvent <sup>b</sup>	$\lambda_{\text{em}}$ , nm
<b>3</b>	THF	345	75850	TFA/THF(2:8)	320	TFA/ THF(2:8)	380
	THF	468	37120	TFA/THF(2:8)	467		
	THF	675	1050	TFA/THF(2:8)	534		
<b>5</b>	THF	344	72500	TFA/THF(2:8)	321	TFA/ THF(2:8)	380
	THF	462	34900	TFA/THF(2:8)	466		
	THF	673	1015	TFA/THF(2:8)	534		

<sup>a</sup> $\lambda_{\text{ex}}$  (excitation wavelength) = 295 nm for **3** and **5**. <sup>b</sup> TFA/THF = v/v.

Upon protonation (by addition of 5%, 10%, 15% and 20% trifluoroacetic acid) of copolymers **3** and **5** in THF in electron spectra, new absorption bands with  $\lambda_{\text{max}}$  at 534 nm were observed (Table 1), characteristic of azulene cations **3-H<sup>+</sup>** and **5-H<sup>+</sup>**. The maxima of their absorption showed the significant redshift of 182 nm compared to the cation of the initial azulene **1** ( $\lambda_{\text{max}}$  352 nm [13]), due to the expansion of the  $\pi$ -conjugation in the five-membered ring by fluorene fragments. In addition, the isobestic points were observed in these spectra at 495 and 496 nm (Figure 4), suggesting the presence of convertible copolymers: neutral **3/5** and protonated **3-H<sup>+</sup>** / **5-H<sup>+</sup>**.

Discoloration of copolymers **3** and **5** was also observed when different amounts of TFA were added. Unlike the dark blue color of the initial azulene **1**, the tetrahydrofuran solutions of copolymers **3** and **5** had the yellowish green color. Adding different concentrations of TFA to the copolymer solution resulted in color change from light orange to dark green (Figure 4). Treatment of protonated solutions **3** and **5** with triethylamine resulted in recovery of the starting state, which indicated the reversible protonation-deprotonation reaction.

It is known that compared to colorless naphthalene, the color of azulene **1** is caused by weak repulsion energy between electrons on practically orthogonal HOMO and LUMO. It is also known that substitutions at the C<sub>1</sub> or C<sub>3</sub> atoms of azulene significantly change the electronic structure and optical properties (Wang, et al, 2003). Therefore, the color changes of **3** and **5** are closely related to the HOMO and LUMO state changes of azulene links induced under protonation conditions.

Thus, TFA doping of polymers **3** and **5** results in the formation of polymeric azulonium cations, with subsequent changes in their electronic spectrum and color.

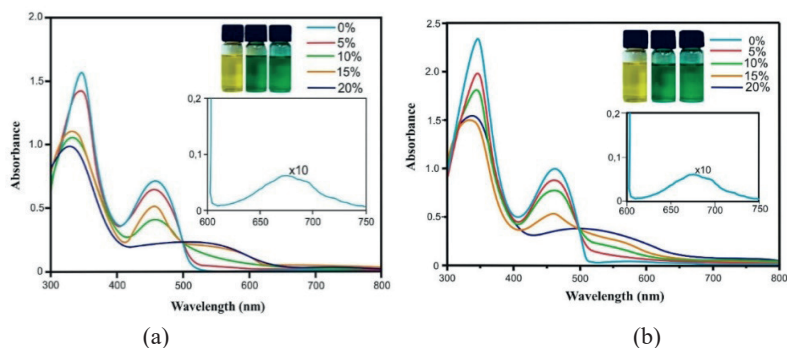


Figure 4. UV-visible spectra of (a) **3** and (b) **5** ( $C=3 \cdot 10^{-5}M$  in THF; in the presence of trifluoroacetic acid).

The fluorescence spectra of copolymers **3** and **5**, including protonated states, are given in Figure 3, and the relevant data are tabulate (Table 1). As shown, the spectra lack emission bands **3** and **5** in THF (Figure 5). However, when TFA was added (i.e., 5%, 10%, 15%, 20%), the solutions of copolymers **3** and **5** emitted light with  $\lambda_{em}$  at 380 nm (Table 1). As can be seen from the fluorescence spectra, the emission intensity of the copolymers varied depending on the TFA concentration. Thus, the intensity of radiation **3** and **5** reached its highest level at 20% concentration of TFA. However, further increases in TFA concentration resulted in a drop-in fluorescence intensity. This was probably due to strong self-absorption or the presence of unprotonated azulene fragments in the polymer backbone, which also led to the decrease in radiation intensity.

As described above, the abnormal anti-Kasha fluorescence of azulene from state  $S_2$  instead of  $S_1$  was due to the large energy gap  $S_1-S_2$ . Obviously, under the conditions of protonation of copolymers **3** and **5** TFA with the subsequent formation of azulenyl cations, a noticeable change in the energy levels  $S_1$  and  $S_2$  occurs, as well as the decrease in the gap between HOMO and LUMO. The following factors obviously affect the decrease in the HOMO-LUMO energy gap: 1) the presence of azulenyl cations in the system and 2) the decrease in the degree of delocalization of  $\pi$ -electrons in the five-membered azulene ring.

Therefore, it can be argued that the «launching» of the radiation in **3-H**<sup>+</sup> and **5-H**<sup>+</sup> copolymers is the consequence of the significant change in the energy of the frontal molecular orbitals HOMO-LUMO.

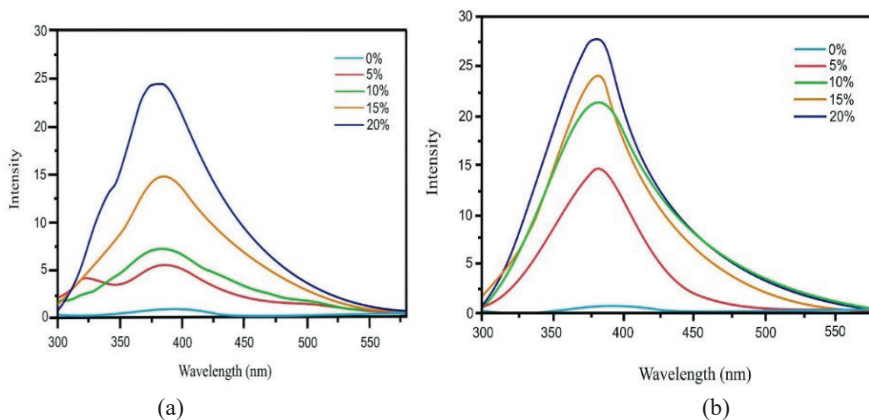


Figure 5. Fluorescence spectra of (a) **3** and (b) **5** ( $C=3 \cdot 10^{-5} M$  в THF; in the presence of trifluoroacetic acid). The excited wavelengths ( $\lambda_{ex}$ ) are 295 nm.

## Conclusion

In conclusion, fluorene-azulene  $\pi$ -conjugated copolymers were synthesized using the Suzuki-Miyaura cross-coupling reaction with high yields: poly[2,7-(9,9-dioctylfluorenyl)-alt-(1',3' - azulenyl)] **3** and poly[1,3-bis(9',9'-dioctylfluoren-2'-yl) azulenyl]-alt-[1'',3'' - azulenyl] **5**. Copolymers are nonfluorescent, because of the existence of azulene units in the system, which suppress radiation. However, in the presence of trifluoroacetic acid, these copolymers became fluorescent with emission maxima ( $\lambda_{em}$ ) at 380 nm. The formation of azulenyl cations in **3-H<sup>+</sup>** and **5-H<sup>+</sup>** copolymers has been shown to significantly affect fluorescence activation. It has also been found that solutions of the obtained copolymers in THF significantly change color when different amounts of trifluoroacetic acid are added.

The results obtained demonstrate that the introduction of azulene units into the polymer backbone has great potential for the development of new proton functional materials.

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