

New nanoporous polyhetroarylenes with ultra-low dielectric permittivity

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Due to constantly increasing need for creating and the miniaturization of VLSI the development of polymers with ultra-low dielectric permittivity becomes more and more required. Such polymers should combine the low dielectric permittivity with high thermal stability and mechanical properties [1]. At present fluorinated PI ($\epsilon=2.6-2.8$) correspond to most of the requirements, which are set for polymer dielectrics [2].

Nevertheless such requirements are becoming more and more severe and next generation materials should possess $\epsilon < 2$ which is a good stimulus for further search of dielectrics with ultra-low dielectric constants [3].

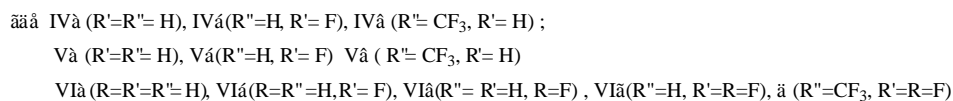
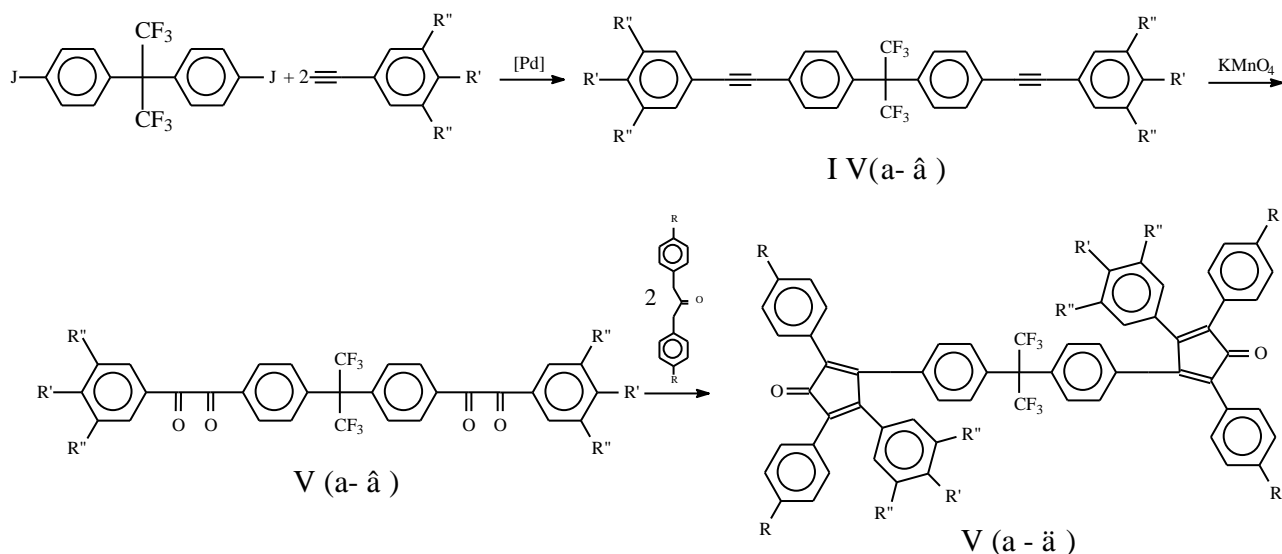
One of the approaches to make the polymer materials with ultra-low dielectric constant is the use of thermally stable polymer micro- and nanoporous structures. The latter ones are usually obtained from micro-phase separated block-copolymers, the pores are formed during heating, when the thermally non-stable blocks are decomposed. Never the less such approach is very effort consuming and is followed by some unwished side effects. In the search for polymers with ultra-low ϵ a number of research groups studied the methods to make a microporous media in polymer matrices, basing on two principles:

1. Thermal decomposition of block-copolymers, containing the thermally stable and unstable block [4,5];
2. Thermolysis of polymer mixtures with high Tg and thermally unstable fillers [6].

We proposed effective and totally different method of making pores in the polymer matrix. The method is simpler, original and ecological and is based on the evaporation of super-critical carbon dioxide. In contrast to other methods the supercritical CO₂ allows the unique ability to approach the lowest possible ϵ' by means of choice of molecular structure of polymers and regulating the micro- and nanopore size.

The purpose of given work is the design and synthesis of fluorinated phenyl-substituted polyphenylenes and following development of materials with ultra-low dielectric constant ($\epsilon' < 2$) in combination with high thermal and mechanical parameters in supercritical CO_2 and the search for dependence of ϵ' from the structure of macromolecules and the shape and shape of the pores. The improvement of dielectric parameters of polymers will be accomplished in two stages A. Using the synthesis of fluorinated phenyl-substituted polyphenylenes ($\epsilon \sim 2.6$) which is much lower than phenylsubstituted polyphenylenes and PI; B. Making the micro- and nanoporous structures ($\epsilon' = 1.6$) in the matrices of the abovementioned polymers in supercritical CO_2 . The advantage of proposed polymer systems consists in the fact, that they possess high hydrophobicity, large free volume, absence of polar groups and as a consequence have ultra-low dielectric constant ($\epsilon = 2.2-2.5$) in combination with high thermal stability and good processibility. Advantage of proposed method to obtain the nanoporous materials in conditions of super-critical CO_2 is the ecology and simplicity of the method.

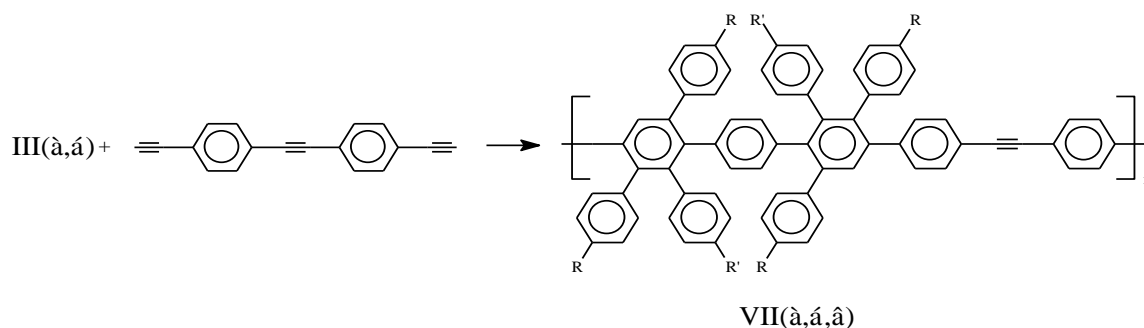
As a consequence, in the frame of given research the synthesis of new fluoro-containing bis-tetraarylcyclopentadienons (BTACPD)- monomers, used for the obtaining of phenyl-substituted polyphenylenes (PSPP) basing on Dils-Alder reaction. The substances contain fluorine in hexafluoroisopropylidene central groups or in phenyl cycles. The synthesis of BTACPD, containg fluorine in phenyl rings was accomplished using standard method, including Pd-catalyzed cross-coupling of dihaloidarylenes with 2-fold molar amounts of phenylacetylene or 4-fluorophenylacetylene, analogous to the following oxidation of so-obtained bis-(phenylethynyl)arylenes and bis-(4-fluorophenylethynyl) – arylenes by potassium permanganate up to bis-(phenylglyaxalyl)arylenes and bis-(4-fluorophenylglyaxalyl)-arylenes and the transition of the latter into BTACPD under the action of 1,3-diphenylacetone or 1,3-(4-fluorophenyl)-acetylene(Scheme 1)



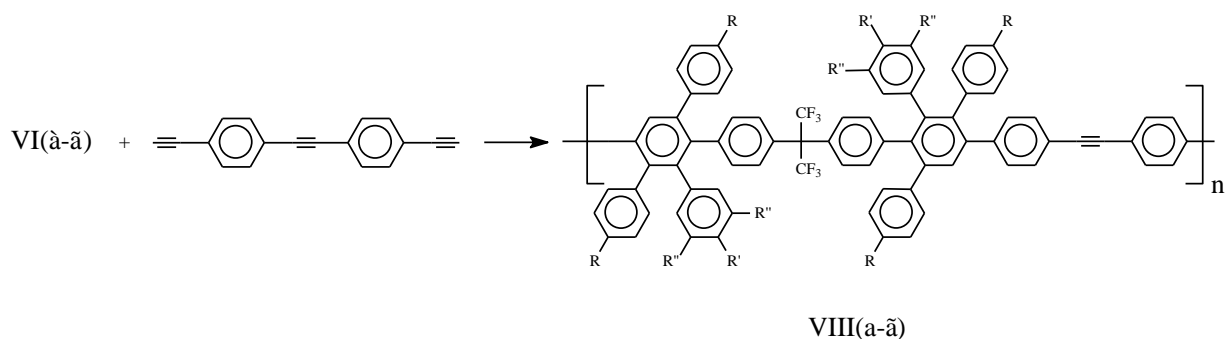
We should note here, that the intermediates, obtained during BTACPD synthesis (Scheme 1,2) and namely diethynylenes (I),(IV) and bis-(α -diketones)(II),(V), are of great importance as the monomers for the synthesis of different types of polymers. Thus, substances I (a-b) and IV(a-b) can be used to obtain phenyl-substituted polyphenylenes in conditions of Dils-Alder reaction and also cross-linked polyphenylenes; substances II(a-b) and V(a-b) can be used in synthesis of polyquinoxalines, poly-ass.-triazines and polyaryletherketones. The composition and structure of intermediates I,II,IV,V and goal products III,VI confirmed by elemental analysis data (Table 1) and also IR and CS spectroscopy(Table 2). According to the data, presented in Table 1, all intermediates and goal products were obtained with high yield and elemental analysis data correspond to calculated values. The synthetic route of obtaining the goal products III and VI was controlled by IR, FTIR – spectroscopy(Table 2). In particular, CS-spectra of substances I and IV, in the range $\lambda=2210-2221\text{ cm}^{-1}$ possess strong absorbance lines, typical for acetylene (C \equiv C) and weak peaks in the range $\lambda=1661-1683\text{ cm}^{-1}$, typical for walent vibrations of CO groups in α -diketone fragments of substances II and V. The absence of signals in the range $\lambda=2210-2221\text{ cm}^{-1}$ confirm full conversion of ethynyl group back into α -diketone. In IR spectra of goal products III and VI the peaks $\lambda=1709-1714\text{ cm}^{-1}$ appear,

which are typical for valent vibrations of carbonyl group of tetraphenylcyclopentadiene fragment, and the bands $\lambda=1661-1683\text{ cm}^{-1}$ disappear, which are marks of α -diketonate groups. The structure of intermediates I,II,IV,V and goal products III,VI also confirmed by NMR ^1H , NMR ^{13}C and NMR ^{19}F . In proton spectra of Ia and Ib. we can see two multiplets at $\delta = 7.46-7.52$ (m, 4H), $7.19-7.24$ (m,6H) и $7.44-7.54$ (m, 8H), 7.05 (t, 4H) m.d. correspondingly. In NMR spectra NMR ^1H bis- α -diketones IIa and IIb signals are shifted into weaker field in comparison to diethynyl intermediates I due to induction effect of electron-acceptor CO-CO groups and reflect in the range $\delta=8.10 -7.52$ and $8.15 -7.47$ m.d. The distinctive feature of NMR ^1H spectra of compounds I and II is the presence of intensive signals, corresponding to central benzene fragment. In NMR ^1H spectra of compounds IVb and Vb in aromatic part at $\delta = 7.57, 7.53,$ and $7.99, 7.52$ there are two doublets and two multiplets at $\delta = 7.51, 7.05$ and $8.03,7.21$ m.d., which well correspond to proposed structures. Proton spectra IV and Vb in aromatic part at $\delta = 7.57, 7.53,$ and $7.99, 7.52$ are two doublets and two multiplets at $\delta = 7.51, 7.05$ and $8.03, 7.21$ m.d., which well correspond to proposed structures. Proton spectra of IVb and Vb are well resolved: besides two singlet pairs at $\delta = 7.96, 7.83$ and $8.46, 8.10$ m.d. are also two doublets at $\delta = 7.57, 7.41$ and $8.07, 7.59$ m.d. correspondingly. The relation of signals in NMR ^1H was made according to relation of integral intensities of signals and basing on values of spin-spin interaction. In NMR ^{13}C of bis-ethynyl compounds I and IV in the range $\delta = 80-90$ m.d. in all cases there appear two intensive signals, corresponding to acetylene fragments, in case of compound IV in all cases in aliphatic part at $\delta = -63.11 - 63.32$ m.d. appear the signals, belonging to quarternized carbon in hexafluoropropylidene fragment, which is split into septet due to two CF_3 groups. In aromatic part of compound IV in all cases the quartet in the range $\delta = 119-125$ m.d. appears, corresponding to C atom of CF_3 group. Besides abovementioned signals in NMR ^{13}C spectra of bis-ethynyl compound of IVb the doublet at $\delta = 163.85 - 161.43$ m.d., typical for C atom directly connected to fluorine atom with the split constant $J=257.1$ Hz. In carbon spectra of compounds II and V in all cases in the range $\delta = 189.30-193.95$ m.d. besides the above mentioned signals two other peaks appear, belonging to two non-equivalent carbonyls bis- α -diketone fragment, and the absence of signals in the range $\delta = 80-90$ confirms full conversion of ethynyl group into α -diketon. Besides all mentioned typical signals

between $\delta = 100$ and 160 m.d., in those spectra also reflect themselves intensive signals, corresponding to different types of carbon atoms, which well coincide with proposed structures. In NMR ^{19}F spectra of compounds Ib and IIb represent themselves signals at $\delta = -110.44$ and -101.21 m.d. typical for fluorine atoms, placed in para- position to acetylene and bis- α -diketon groups. In NMR ^{19}F compounds IV and V are in the ranges $\delta = -62.94 - -63.35$, -109.85 and -100.14 m.d. corresponding to CF_3 group and Ar-F. Proton spectra of bis-cyclons III and VI are complex enough and reflect in weak fields as multiplets in the range $\delta = 6.90-7.77$ m.d. More informative are the spectra NMR ^{13}C . Typical peculiarity of NMR ^{13}C spectra of compounds III and VI is the presence of weak field signals in the range $\delta = 200.31-199.11$ m.d., typical for carbonyl group of cyclopentadiene fragment. In all cases the carbon spectra of bis-cyclones VI, containing hexafluoroisopropylidene groups in the range $\delta = -63.75 - -63.18$ m.d. appears the signal, which is split into septet due to CF_3 groups. In case of VIb besides above mentioned signals in aromatic part of carbon spectra at $\delta = 164.41 - 161.09$ m.d. there appears a doublet, typical for carbon atom, directly connected with fluorine atom. For the compounds Vc and VIg at $\delta = 164.04-163.95$, $160.75-160.67$ and $164.48-164.38$, $164.02-163.93$, $161.15-161.06$, $160.78-160.68$ m.d. two and four doublets appear, directly corresponding to carbon atoms, which are connected to two and four non-equivalent fluorine atoms. FFPP was obtained by reaction of biscyclopentadienes with bisethynyl compounds according to



VIIa(R=H, R'=F); VIIb(R=F, R'=H); VIIc(R=R'=H)



VIIIa(R=R'=R''=H), VIIIb(R=R''=H, R'=F), VIIIc(R''=R'=H, R=F), VIId(R''=H, R'=R=F)

FFPP was synthesized in 1,2,4-trichlorobenzene at 150° C during 40 hours. All reactions were taking place homogeneously with yields close to quantitative ones, viscous properties are in the range of 0.51-0.85 dl/g. MM and polydispersity of polymers determined by GPC are varied in the range $M_w = (86.7-120.0) \times 10^3$, $M_n = (30.1-50.4) \times 10^3$ and 1.98-2.88 correspondingly. The structure of FFPP was confirmed by IR, NMR ^1H , NMR ^{13}C and NMR ^{19}F spectroscopy data. In IR spectra of all polymers the maxima of absorbance of ethynyl groups ($3210, 2200\text{cm}^{-1}$) and carbonyl fragments of tetraphenylcyclopentadienons (1709cm^{-1}), which points to the formation of polymer and evidences the low concentration of tail groups. In NMR ^{13}C spectra also reflect themselves in ranges $\delta=200$ and 80 m.d., which are typical for cyclopentadiene and terminal acetylene groups. In NMR ^{19}F spectra of polymers VII(a-c) the signals at $\delta= -112$ m.d. are present, which are typical for fluorophenyl substitutes of polymers. All synthesized polymers are soluble in common organic solvents – toluene, TGF, DMSO, DMFA, NMP and chlorophorm. Good solubility in chlorophorm allowed to obtain films with the tensile strength in the interval of 58.8-69.5 MPa and relative

elongation $\epsilon' = 3.1-8.1$ (Table 3). FFPP exhibit high values of glass transition temperatures in the range 239-340 C (Table 4). The results of TGA analysis show excellent thermal stability of FFPP. The temperature of 10% mass loss for all polymers in the Argon atmosphere and in air is in the range 500-521 C and 417-476 C correspondingly (Table 3). The values of initial ϵ' for FFPP in vacuum at 1 kHz was varied in the range 2.54-2.74. It is lower than ϵ' in comparison with usual FPP (see VIIc $\epsilon' = 2.8$). The introduction of fluorine into the macromolecules of FFPP was followed, as expected, by lowering of dielectric constants of those polymers in comparison with previously synthesized non-fluorinated FPP. It is due to, most probably, to the exchange of hydrogen by bulky fluorine atoms, which lower the electron polarizability of C-F bond in comparison with C-H, lowering of packing density of chains, increase of free volume and hydrophobicity of the polymer.

On the second stage on the basis of obtained FFPP the micro- and nano- foams with controlled size and shape of pores were synthesized. Initially the polymer films were saturated with carbon dioxide in the reactor under saturation pressure ($P_{\text{sat}} = 5 \text{ MPa}$) and temperature of saturation ($T_{\text{sat}} = 25^\circ \text{C}$). After fast evacuation of CO_2 from reactor, the film was placed into glycerol bath for 30 seconds, which was heated up to T_{foam} (240-290 $^\circ \text{C}$), the intense desorption from the surface and simultaneous foaming of polymer material took place. The CO_2 content at the surface was decreased and dense "skin layer" was formed wrapping foamed inner nucleus. Afterwards, the foamed samples were placed into the mixture of ethanol with water (1:1) and dried under vacuum during 1 day at 30 C in order to eliminate the traces of water and ethanol. Morphology of obtained films was mainly depending on P_{sat} , T_{sat} and T_{foam} .

The properties of foamed polymers, T_g , density and ϵ' are seen from Table 4. The porous films of FFPP were exhibiting the microcellular morphology according to SFM data. Main reason is in high solubility of CO_2 in FFPP, which is determined by formation of gas bubbles of high density. Porosity of 40% was reached for all amorphous polymers at their maximum temperatures of foaming. Depending on the structure of polymers and foaming temperature all obtained materials demonstrate significant decrease of ϵ' in the limits of $\epsilon' = 2.74-1.61$. In conclusion it should be noted that T_{sat} can be used as an additional parameter for regulating the pore size. The decrease of T_{sat} down to zero

significantly increases the solubility of CO₂ and at the same time leads to significant decrease of diffusion of CO₂ from the film. Microcellular morphology prevail, if the saturation takes place at 25 C, at the same time nanoporous morphologies, which possess remarkably low ϵ' are obtained at T_{sat}= 0 °C. Only nanoporous structures with the pore size less than 10 nm can potentially be applied in multi-layer electronic devices. Further research of the influence of T_{sat} on the size and shape of pores and ϵ' is planned by our group.

CONCLUSIONS

We synthesized and investigated a number of new fluorine containing FPP, which possess high thermal stability, hydrophobicity, high free volume, and hence low dielectric permittivity ($\epsilon' \sim 2.74 - 2.54$). The polymers also possess good processibility and film-forming properties.

Relating to the synthesized FPP the process of micro- and nano- porous structures formation in the supercritical CO₂ was studied.

As a result of directed study based on the obtained data new thermally stable, film-forming polymer materials with ultra-low controlled dielectric permittivity ($\epsilon' = 1.97-1.61$) were obtained.

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Table 1. Some parameters of intermediates and goal products I-VI

№	Tm., °C	Brutto-formula	Elemental Analysis			Yield, %
			Calculated / found, %			
			C	H	F	
Ia	184-186	C ₂₂ H ₁₄	<u>94.93</u>	<u>5.07</u>	-	81
			94.67	4.98		
Iб	191-192	C ₂₂ H ₁₂ F ₂	<u>84.06</u>	<u>3.85</u>	<u>12.09</u>	78
			83.92	3.69	11.91	
IIa	125-127	C ₂₂ H ₁₄ O ₄	<u>77.18</u>	<u>4.12</u>	-	68
			77.11	4.22		
IIб	223-225	C ₂₂ H ₁₂ F ₂ O ₄	<u>69.84</u>	<u>3.20</u>	<u>10.09</u>	67
			69.43	3.11	9.65	
IIIa	312-314	C ₅₂ H ₃₀ F ₄ O ₂	<u>81.88</u>	<u>3.96</u>	<u>9.96</u>	87
			82.13	3.73	9.71	
IIIб	325-327	C ₂₂ H ₃₂ F ₂ O ₂	<u>85.95</u>	<u>4.43</u>	<u>5.23</u>	83
			85.67	4.26	5.00	
IVa	90-91.5	C ₃₁ H ₁₈ F ₆	<u>73.81</u>	<u>3.60</u>	<u>22.60</u>	89
			73.66	3.58	22.34	
IVб	176-178	C ₃₁ H ₁₆ F ₈	<u>68.89</u>	<u>2.98</u>	<u>28.14</u>	92
			68.59	2.87	28.00	
IVв	115-117	C ₃₅ H ₁₄ F ₁₈	<u>54.14</u>	<u>1.80</u>	<u>44.04</u>	81
			54.09	1.75	43.65	
Va	145-147	C ₃₁ H ₁₈ F ₆ O ₄	<u>65.66</u>	<u>3.19</u>	<u>20.05</u>	95
			65.20	2.15	19.61	
Vб	111-113	C ₃₁ H ₁₆ F ₈ O ₄	<u>61.59</u>	<u>2.66</u>	<u>25.14</u>	88
			61.33	2.42	24.69	
Vв	115-117	C ₃₅ H ₁₄ F ₁₈ O ₄	<u>50.02</u>	<u>1.68</u>	<u>40.69</u>	81
			49.87	1.61	40.31	
VIa	236-238	C ₆₁ H ₃₈ F ₆ O ₂	<u>79.89</u>	<u>4.17</u>	<u>12.43</u>	87
			79.58	4.19	12.21	

VIб	178-180	$C_{61}H_{36}F_8O_2$	<u>76.88</u> 76.70	<u>3.80</u> 3.94	<u>15.95</u> 15.71	82
VIв	220-222	$C_{61}H_{34}F_{10}O_2$	<u>74.09</u> 73.91	<u>3.70</u> 3.46	<u>19.21</u> 18.94	91
VIг	229-231	$C_{61}H_{32}F_{12}O_2$	<u>71.49</u> 71.09	<u>3.15</u> 3.21	<u>22.24</u> 21.73	95
VIд	145-147	$C_{65}H_{34}F_{18}O_2$	<u>65.66</u> 65.20	<u>2.88</u> 2.93	<u>28.76</u> 28.41	95

Table.2. Spectral parameters of intermediates and goal compounds I-VI

№	Spectra NMR, δ , м.д.			
	КР, ν ,	1H	^{13}C	^{19}F
IIa	1671 CO	8.10(4H),7.96(4H))7.68(2H),7.52 (,H	193.32(CO),193.17(CO),137.04(ч),135.17, (ч), 130.16,129.88, 129.06 132.48	–
IIб	1668 CO	8.15 (4H), 8.09 (8H),7.47(т,8H	192.44 (CO), 192.10 (CO), 167.13, 161.56, 134.24, 132.51,115.99, 115.78	-101.21
IIIa	1709 CO	6.60-8.10 (м, 30H)	200.31,165.05,162.41,151.93,151.55,137.58,137.26,1 32.45,132.31,130.65,127.10,126.74, 125.33, 116.57,	-112.37, -113.05
IIIб	1711 CO	7.30-7.72 (м, 32H)	199.21,164.53,161.95,159.33,152.29,137.33,135.08,1 33.58,132.86,132.74,132.68,126.95,126.15,125.61, 125.31, 115.37, 115. 09	-113.15
IVa	2212 $C\equiv C$	7.20- 7.56 (м, 18H);	136.19,134.32,131.27,130.18,129.63,129.15,128.83,1 28.31,127.11,123.61,121.55,116.12,82.17,93.95,65.8 8,65.56,65.31,65.06,64.81,64.56, 64.31	-63.11
IVб	2221 $C\equiv C$	7.50-7.54 (м, 8H), 7.37 (д, 4H), 7.05(т, 4H)	163.85,161.35,133.48,133.17,132.87,131.24,130.07,1 28.14,125.27,124.14,122.43,119.58,118.78,115.53,11 5.07,94.89,87.73,65.10, 64.85, 64.60, 64.35, 64.09,	-109.85 -63.32
IVв	2210 $C\equiv C$	7.96 (с, 4H), 7.84 (с, 2H), 7.57 (д,	133.78,132.49,132.15,131.81,131.51,131.45,130.23,1 25.01,124.15,123.02,121.86,121.43,87.8,91.20,	-63.11 ($12CF_3$)-

		4H), 7.42 (д, 4H)	65.78, 61.16, 65.16, 62. 19, 64. 49, 62.67, 63.87	63.54
Va	1681	7.55 (т, 8H); 7.67 (т,2H), 7.99 (т, 8H)	193.95,193.58,138.17,135.74,133.07,132.08,130.89,130.14,129.89,129.54,128.01,124.91,122.05,118.02, 66.88, 64.06, 66.18, 63.16, 65. 47, 62 .66, 64.77	-63.55 (CF ₃)
Vб	1683	7.20 (т, 4H), 7. 52 (д, 4H), 7. 96-8.05(м, 8H)	192.43,191.64,168.13,165.56,116.53,116.31,132.81,132.72,127.68,133.19,129.48,130.73,139.04,127.68,124.83,121.96,119.10,66.88,64.06,66.18,63.16,	-62.94 (2CF ₃)- 100.14
Vв	1661	7.5(4H),8.0(4H),8.1(2H),8.4 (4H)	190.32,189.30,139.62,134.20,133.07,132.72,130.94,130.05,129.90,129.77,126.61, 123. 87, 121.16, 118.45,	-63.01 -63.18
VIa	1710	7.31-7.13 (22H), 7.18 - 7.1 (8H), 6.9 - 6.9 (4H), 6.85 (4H)	199.11,154.01,152.87,134.67,132.97,132.60,130.36,130.16,129.97.129.59,129.12,128.97,128.61,127.99,127.86,127.69,127.51,125.75,125.05,123.74), 64.22	-63.45 (CF ₃)
VIб	1714	7.59 - 6.80 (м. 36H)	199.77,164.41,161.09,153.03,152.61,134.31,133.22,131.36,131.26,130.35,130.09,129.86,129.76,129.32,129.15,128.74,128.63,128.23,128.16,127.93,127.81,127.25,127.09,126.09,125.43,115.45,115.16, 64.89	-63.75 (CF ₃), -111.23 (F)
VIв	1710	6.90-7.06(17H) 7.20-7.33(17H)	199.92(сo),164.04,163.95,160.75,160.65,154.03,152.84,134.11,133.28,132.50,131.90,131.79,129.86,129.17,129.05,128.04,128.15,126.44,126.39,126.27,126.22,124.87,124.19,115.49,115.45,115.20,115.17,65.07, 64.70, 64.38, 64. 38,.	-63.64 (CF ₃) 112.69 (F) 113.04
VIг	1716	6.80-7.10 (м, 20 H), 7.26 -7.40 (м,12H),	199.65,164.48,164.02,161.15,160.78,152.91,152.44,134.12,133.36,132.55,132.41,131.88,131.78,131.28,131.17,130.71,130.00,129.59,129.05,128.46,126.26,126.11,125.05,124.37,115.90,115.59,115.50,115.30, 115.21, 114.90,.	-63.64 (CF ₃), -110.7 -112.40 -112.73
VIд	1714	8.2-7-29 (м, 34 H),	199.28,158.32,156.77,140.56,139.14,138.14,135.11,128.21,128.01,127.77,126.65,126.14,125.81,125.32,120.73,120.57,120.32,117.45,114.61,111.75, 64.73, 64.45, 64.18, 63.89, 63.61, 63.33,	-63.18 (CF ₃), -63.58 (CF ₃),

Табл. 3. Some parameters of FPP

Polymer	$\eta_{\text{прив.}}$ (МР, 25°C) дл/г	M_n	M_w	M_w/M_n	$T_{10\%}, ^\circ\text{C}$		Film properties at stretching	
					air	Argon	σ , МПа	ε , %
VIIa	0.78	50.4	99.8	1.98	470	510	67.0	5.0
VIIб	0.85	48.3	101.4	2.10	477	521	69.5	5.4
VIIIa	0.64	45.0	112.5	2.50	473	513	65.8	8.1
VIIIб	0.59	39.3	110.1	2.80	449	501	66.1	7.8
VIIIв	0.70	50.0	120.0	2.40	476	520	63.2	7.0
VIIIг	0.51	30.1	86.7	2.88	417	500	58.8	3.1

Table 4. Some electrophysical properties of FPP VII и VIII

Polymer	$P_{\text{sat.}}$, МПа	$T_{\text{sat.}}$, °C	$T_{\text{foam.}}$, °C	T_g , °C	Density, g/cm^3	Morphology	ε' [a]
VIIв	5.0	25	-	340	1.28	dense	2.80
			250		1.22	micropores	2.58
			290		0.98	nanopores	2.00

VIIa	5.0	25	- 250 290	330	1.25 0.95 0.78	dense micropores nanopores	2.74 2.4 1.77
VIIб	5.0	25	- 250 290	311	1.24 0.91 0.71	dense micropores nanopores	2.69 2.18 1.70
VIIIa	5.0	25	- 240 280	297	1.24 1.08 0.86	dense micropores nanopores	2.63 1.97 1.68
VIIIб	5.0	25	- 240 280	239	1.24 1.10 0.71	dense micropores nanopores	2.58 1.91 1.65
VIIIв	5.0	25	- 240 280	303	1.24 1.02 0.70	dense micropores nanopores	2.54 1.88 1.65
VIIIr	5.0	25	- 240 280	259	1.23 0.98 0.73	dense micropores nanopores	2.54 1.81 1.61

[a] – measured at 1 KHz at, 25 °C and vacuum 10^{-4} mm. Hg