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Spectroscopic study of liquid crystals in confined volume

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Abstract

IR absorption spectra of nematic liquid crystal, pentylcyanobiphenyl (5CB), dispersed in porous glass and molecular sieves of types Si, Al–MCM-41 and Cu–Si, Al–MCM-41 (diameter of pores is about 40 Å) have been investigated in the region of fundamental and overtone vibrations. Mechanism of interaction between a part of 5CB molecules and active centers of porous interface was estimated. In this case, enough strong interaction between the cyano groups of 5CB and hydroxyl groups of the channel surface is realized. It was also shown that another part of the 5CB molecule exists in the liquid crystal state and its amount depends on the size and nature of the pores. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Liquid crystal; Confinement; IR spectra; Interface interaction

1. Introduction

In recent years, great attention has been devoted to the study of intermolecular interactions and physical properties of different heterogeneous liquid crystal (LC) systems due to their wide application in electronic devices. These may be either filled nematic LCs, namely LC + Aerosil systems (inorganic particles with a diameter of about 100 Å and concentration of 0.2–10 wt.% forming H-bonded associates) or confined LCs dispersed in inorganic porous matrices (porous glasses (PGs) or mesoporous molecular sieves with channels of 20–2000 Å diameter). LCs in these systems manifest new interesting physical properties that are absent in bulk materials [1–7].

The investigation of such systems is also useful in establishing the interaction mechanism of LCs with surface active centers. Although great success was achieved in the understanding of physical properties of LCs in various porous matrices, little work has been done to characterize the interaction of LC molecules with a pore surface on molecular level.

It is known that IR spectroscopy is a sufficiently sensitive method for the identification of local surrounding and changes in the intermolecular interactions. Therefore, it may be used as a powerful tool for studying the processes of interaction between the LC molecules and active centers of inorganic fillers or matrices.

Earlier we have studied the molecular interactions in LC–Aerosil systems by IR method and shown that the hydrogen bonds between the 5CB molecules and

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the aerosil particles play an important role in the "memory" effect [2,8].

In the present work, we report the results of the investigation of vibrational spectra of nematic LC-pentylcyanobiphenyl (5CB) included in porous glasses and in different types of molecular sieves (with the pore diameter of 40 Å). The study is aimed at establishing the character of interaction between 5CB molecules and interface active centers of porous inorganic matrices.

2. Experimental

2.1. Objects of investigation

4-Pentyl-4′cyanobiphenyl (5CB), $C_5H_{11}–C_6H_4–C_6H_4–C\equiv N$ (NIOPIC, Russia), that exists in the nematic phase within the temperature range 295.5–308 K, was used as a LC. The structure and spectral features of this substance have been studied in detail [9–11].

Porous silicate glasses with strongly interconnected and randomly oriented pores with an average pore diameter of 40 Å (volume fraction of pores was about 25%) were prepared from the original sodium borosilicate glasses [12]. The matrix framework consists of SiO_4. These porous matrices like porous Vycor glass are characterized by the narrow distribution of pore sizes. In our experiments, the glass plates of 0.2–0.8 mm thickness were used.

Molecular sieves of the MCM-41 type have a unidimensional mesoporous structure with a regular arrangement of hexagonal channels of 40 Å diameter and "free" space diameter of about 25 Å [7]. Both the sieve composition and the physical properties of the compounds included in the sieves are well reproducible. Due to their open porous structure, which allows both the "guest" incorporation into the internal crystal space and their interaction with the active centers, the sieves are suitable for inclusion of large organic molecules [7].

The MCM-41 type mesoporous aluminosilicate molecular sieves Si, Al–MCM-41 (referred to as MCM) studied in this work were manufactured in the presence of cetyltrimethylammonium bromide, $C_{16}H_{33}N(CH_3)_3Br$, the reagent mixture composition was 16.2 SiO_2:Al_2O_3:5.4 Na_2O:13.8 $C_{16}H_{33}N(CH_3)_3Br$: 1677 H_2O. Aluminum sulfate was used as the source of Al supply, which according to Ref. [13] provides the incorporation of total Al into the MCM-41 framework in the tetrahedral coordination. The hydrothermal synthesis was carried out at 150°C during 48 h. According to the chemical analysis results, the SiO_2:Al_2O_3 ratio in the final compound was 15.65 (close to one of the reaction mixture). According to the nitrogen absorption data (ASAP 2000 "Micromerites" device, 77.2 K, activation temperature 573 K), "free" space value of the sieves amounts to 1.16 $cm^3\,g^{-1}$, and their specific surface area is equal to 851 $m^2\,g^{-1}$. The pore diameter distribution (estimated by the Kelvin equation) shows one broad peak at about 35 Å (width ~2Å).

The Cu containing Si, Al–MCM-41 formula Cu–Si, Al–MCM-41 (CuMCM) studied in this work was manufactured by means of ionic exchange in 10^{-2} mol l^{-1} water solution of copper acetate $Cu(CH_3COO)_2$ of air-free specimen at the ratio of solid to liquid phases of 1:100.

2.2. Equipment and methods of preparing the samples

IR spectra of 5CB, porous glasses PG, MCM, CuMCM and heterogeneous systems on their base were measured at room temperature in the 400–4000 cm^{-1} spectral region on UR-20 (Carl Zeiss, Jena) and FTIR Nicolet "Avatar" 360 spectrometers, and in 3500–13 000 cm^{-1} (3000–750 nm) spectral region using UV–VIS–NIR "Cary-5E" (Varian) spectrophotometer. The spectral slit width was 2–4 cm^{-1} in the 400–4000 cm^{-1} region.

For IR studies, the sample of 5CB was prepared as a film between CaF_2 or KBr plates with 15 μm film thickness.

The 5CB–MCM and 5CB–CuMCM heterogeneous systems were manufactured by mixing 50 mg of the molecular sieve with 200 μl of 5CB in the nematic phase. Then the mixture was rinsed with hexane in order to eliminate the residual LC remaining in the channels.

For IR spectra measurements the samples of MCM and CuMCM, as well as heterogeneous systems on their base, 5CB–MCM and 5CB–CuMCM, that were powder-like, were prepared as suspension in

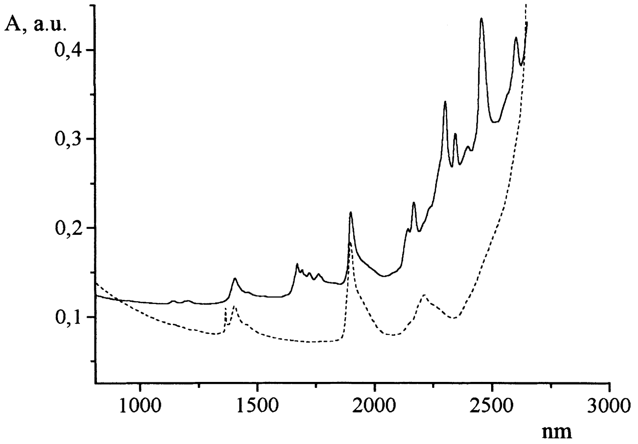

Fig. 1. NIR spectra of the PG (···) and PG + 5CB (—) ($d_{PG} = 0.7$ mm, 300 K).

Nujol or pressed in pellet with KBr to minimize the scattering of infrared radiation.

Before filling, the stabs of the porous glass were heated to 720 K and were endured for 8–10 h. They were then cooled till near room temperature and dipped for 1 h in liquid 5CB at 330 K. IR spectra of porous glass plates (pure and filled) were measured.

The graphical separation of the IR bands into components was performed by using the PEAKFIT computer program.

In Figs. 1–5 the fragments of the spectra of studied samples are presented. Frequencies, relative intensities and assignment of IR bands are represented in Tables 1 and 2.

3. Results and discussion

3.1. IR spectra of 5CB

The IR absorption spectra of alkylcyanobiphenyls in different phases have been studied earlier [2,10]. In the present work, IR spectra of LC 5CB were measured both in the spectral region of fundamental vibration and in near IR (NIR) spectral region, in which overtones and combination modes are located (Tables 1 and 2).

Assignment of the IR bands to normal vibrations of various structural groups was carried out on the basis of literature data. However, it is now difficult to assign

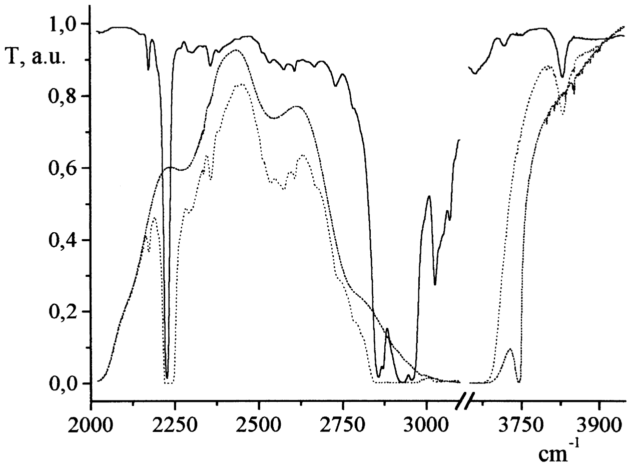

Fig. 2. IR transmittance spectra of the 5CB (—), PG (...) and PG + 5CB (· · ·) ($d_{PG} = 0.7$ mm, 300 K).

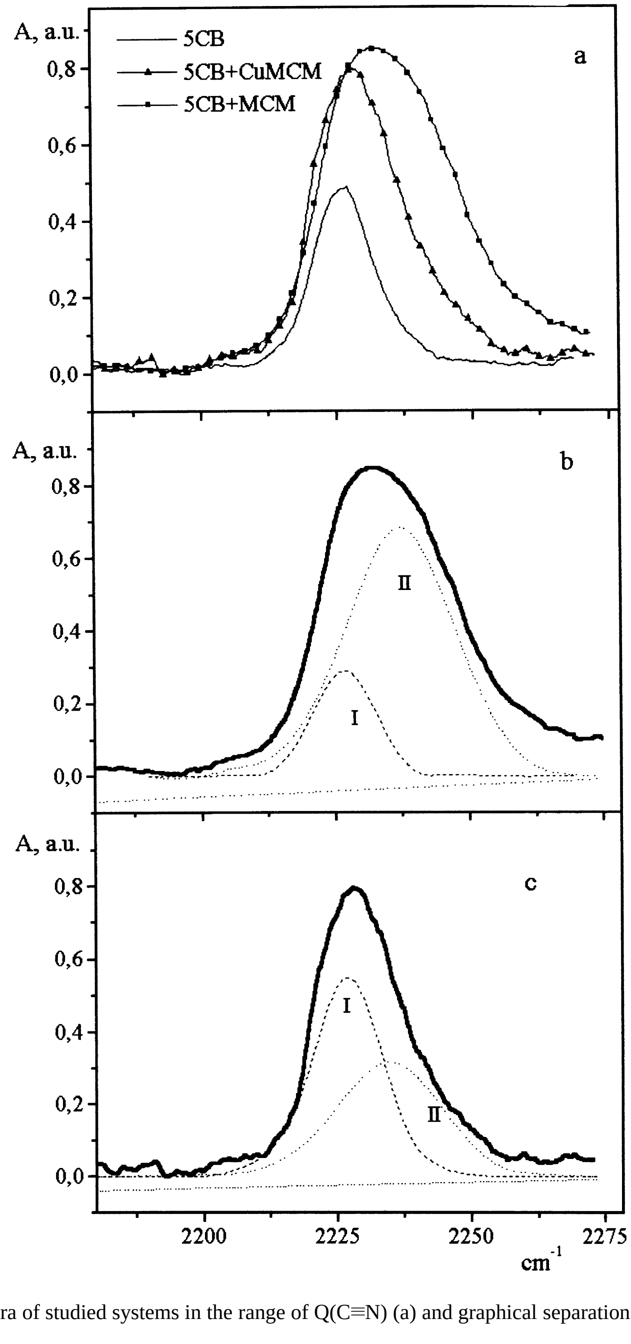

Fig. 3. Fragments of the IR spectra of studied systems in the range of Q(C≡N) (a) and graphical separations of the bands of 5CB + MCM (b) and (c) 5CB + CuMCM.

IR bands in the NIR region, this will be dealt with in a forthcoming paper.

It is known that the dipole moment of 5CB is equal to 4.05 D and is located mainly on the C≡N bond. A detailed study of parameters of the Q(C≡N) band is very interesting. In the liquid crystalline and isotropic phases, the 5CB molecules, unlike those of the other members of the *n*CB and *n*OCB homologous series,

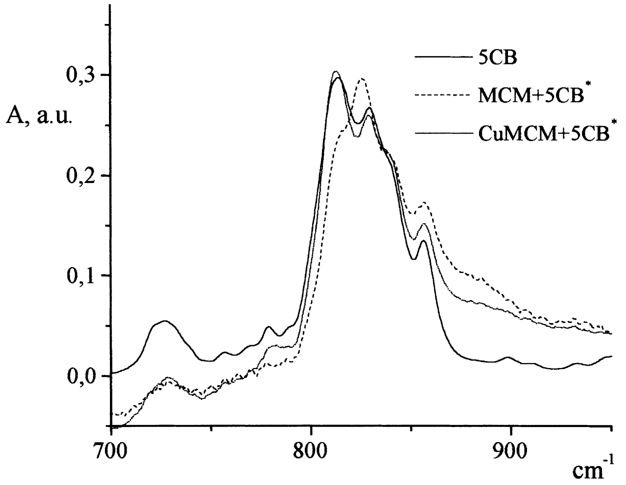

Fig. 4. Fragments of the IR spectra in the range of $\rho(C\overset{\cdots}{-}C\text{–}H)$. * : these spectra are obtained by subtraction of the MCM spectrum.

are arranged in the dimers by the dipole–dipole interactions [14,15]. Isolated molecules of these compounds exist only in the dilute solutions in neutral solvents, giving the characteristic parameters of the Q(C≡N) absorption band: $\nu_{\max} = 2228\,\mathrm{cm}^{-1}$, and $\Delta\nu_{1/2} = 5\,\mathrm{cm}^{-1}$. It should be noted that in the case of the 1:1000 Ar matrix, isolated C_5H_{11}–C_4H_3N–C_6H_4–C≡N (R–C≡N) molecules ($T = 4.2$ K) the Q(C≡N) bandwidth is equal to $4.9\,\mathrm{cm}^{-1}$ (the data were kindly presented by O. Ivanov, Physical and Engineering Institute of Low Temperatures of the National Academy of Sciences of Ukraine). According to our results, the peak position of Q(C≡N) band of 5CB in the nematic phase and the bandwidth are $\nu_{\max} = 2226\,\mathrm{cm}^{-1}$ and $\Delta\nu_{1/2} = 13.2\,\mathrm{cm}^{-1}$, respectively (see Fig. 3 and Table 3).

3.2. IR spectra of porous glasses

There are many very strong IR absorption bands in the PG stab ($d = 0.7$ mm) spectra in the region of the fundamental vibrations. The PG has a transparency “window” in the spectral range 2050–$3000\,\mathrm{cm}^{-1}$ (Fig. 2). It is also transparent in the NIR region ($\nu > 3650\,\mathrm{cm}^{-1}$), where only several weak bands are observed. Three weak broad bands with frequencies

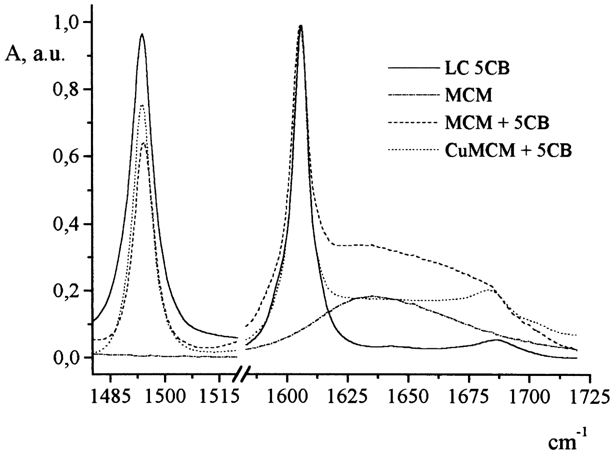

Fig. 5. Fragments of the IR spectra in the range of the $Q(C\overset{\cdots}{-}C)$ vibrations.

Table 1
Frequencies (cm^{-1}) and relative intensities of IR bands of LC 5CB and its systems with MCMs, and their assignment (abbreviations: v, very; s, strong; m, medium; w, weak; sh, shoulder; b, broad; Q,ν—stretching vibrations; δ,γ,β—in-plane and ρ,χ—out-of-plane deformation vibrations; subscripts "s"—is symmetrical, "as"—asymmetrical vibrations)

5CB	5CB + MCM	5CB + CuMCM	MCM	Assignment
	457	457	457	δ (SiO_4) and ν (AlO_4)
543 m	547 m	545 m		
553 m	555 m	553 m		$\gamma(\mathrm{C}{\cdots}\mathrm{C}{\cdots}\mathrm{C})$
566 m	564 m	565 m		
	570–680 wsh	570–680 wsh	570–680 sh w	ν (AlO_4)
637 w	637 w	637 w		
650 w	650 w	650 w		Ring vibration
655 w	655 w	655 w		
725	725	725		ρ (CH_2)
777 w	789 w	783 w		$\rho(\mathrm{C}{\cdots}\mathrm{C{-}H})$
	795 m	795 m	795 m	ν_s bridge bonding SiO_4
812 m	812 m	812 m		
829 m	823 m	827 m		$\rho(\mathrm{C}{\cdots}\mathrm{C{-}H})$, χ (CC)
840	840 w	840 w		
856 w	857 w	857 w		
	943 m	943 m	943 m	SiO_4
1006 m	1006 m	1006 m		$\beta(\mathrm{C}{\cdots}\mathrm{C{-}H})$
	1075 vs	1075 vs	1075 vs	ν_s (SiO_4)
1180 m	1180 w	1180 w		
1186 m	1186 w	1186 w		$\beta(\mathrm{C}{\cdots}\mathrm{C{-}H})$
1285	1285 w	1285 w		
1378 w	1378 w	1378 w		δ_s (CH_3)
1397 m	1397 m	1397 m		$Q(\mathrm{C}{\cdots}\mathrm{C})$
1457 m	1457 m	1457 m		δ_{as} (CH_3)
1466 m	1466 m	1466 m		δ (CH_2)
1494 s	1494 s	1494 s		$Q(\mathrm{C}{\cdots}\mathrm{C})$
1574 w	1574 w	1574 w		$Q(\mathrm{C}{\cdots}\mathrm{C})$
1606 s	1606 s	1606 s		$Q(\mathrm{C}{\cdots}\mathrm{C})$
	1636 mb	1636 wb	1636 mb	δ (H_2O)
1685 vw	1685 sh	1685 sh		Combination tone
2226 s	2226 s	2226 s		$Q_I(\mathrm{C{\equiv}N})$
	2238 s	2236 s		$Q_{II}(\mathrm{C{\equiv}N})$
2857 s	2857 s	2857 s		$\nu_s(CH_2)$
2870 s	2870 s	2870 s		$\nu_s(CH_3)$
2929 s	2929 s	2929 s		$\nu_{as}(CH_2)$
2956 s	2956 s	2956 s		$\nu_{as}(CH_3)$
	3240 w	3240 w	3245 s	
	3400 m	3420 m	3440 vs	ν (OH$\cdots$) in H_2O, Al–OH, Si–OH
	3605 w	3607 m	3600 m	

2300, 2550 and 2790 cm^{-1} are overtones and combination modes of the stretching and deformation vibrations of SiO_4 groups. A very strong broad band with $\nu = 3400\,\mathrm{cm}^{-1}$ and a narrow band with $\nu = 3750\,\mathrm{cm}^{-1}$ are due to stretching vibrations of ν(OH) in the H-bonded associates and "free" OH group, respectively. In the NIR spectra, we observe overtones of these vibrations with a frequency of 7128 and 7326 cm^{-1}.

3.3. IR spectra of Si, Al–MCM-41 and Cu–Si, Al–MCM-41

In the IR absorption spectra of the molecular sieves,

Table 2
Frequencies (cm^{-1}) and relative intensities of IR bands of system with porous glass (PG) in the NIR spectral region (abbreviations as in Table 1)

5CB	PG + 5CB	PG
2174	2174	
2226	2226	
–	2238	
2270	2270 sh w	
2294	2294	
2304	2304	2300 wb
2337 w	2337 w	
2359	2359	
2384	2384	
2443 w	2443 w	
2513 sh	2513 sh	
2537	2536	
	2550 wb	2550 wb
2577	2577	
2610	2610	
2624 w	2624 w	
2670	2670 sh	
2733	2733 sh	
2782	2782	
	2790 wb	2790 wb
	3330 vs b	3400 vs b
		3750 s
3831	3836	
3905 w	3898 sh	
3973	3973	
4068	4067	
4166 w	4167 w	
4263	4263	
4338	4342	
4402 sh	4402 sh	
	4464 sh	4440 sh
	4523 sh w	4523
4572		
4610	4610	
4669	4666	4672 sh
4803 sh	4803 sh	
	5128 sh	5128 sh
	5260	5268
5675	5675	
5807	5807	
5869	5869	
5910	5913	
5986	5986	
6165 w	6165 w	
	6840	6868
	7128	7128
		7326
	8292 w	
	8764 w	

Table 3
Spectral parameters of the Q(C≡N) IR absorption bands. ν_{I} and ν_{II} are the peak positions (cm^{-1}) of components I and II (see Fig. 3), respectively; $\Delta\nu_{1/2}$ is the bandwidth (cm^{-1}), S is the normalized integral intensity (%) (n/m: not measured)

Studied sample	ν_{I}	$\Delta\nu^{I}_{1/2}$	S_{I}	ν_{II}	$\Delta\nu^{II}_{1/2}$	S_{II}
R–C≡N (4.2 K)	2235	4.9	100			
5CB (in solution, 300 K)	2228	5	100			
5CB (LC)	2226	13	100			
5CB + MCM	2226	12	18	2238	25	82
5CB + CuMCM	2226	14	53	2236	22	47
5CB + PG	2226	n/m	n/m	2238	n/m	n/m

the intensive bands of stretching and bending vibrations of various Si(Al)–O aluminosilicate framework are observed in 400–1300 cm^{-1} spectral range (Table 1). In the high-frequency range of the spectra, the broad intensive band of stretching vibrations of H-bonded OH-groups with peak position at $\nu = 3420$ cm^{-1} is observed.

Assignment of their IR bands is listed in Table 1 according to Ref. [16].

3.4. IR spectra of LC 5CB in porous glasses

The formation of new hydrogen bonds between the 5CB molecules and active Si–OH centers of the surface of the pores is proved by the shift of the OH-stretching band $\nu^{\mathrm{ac}}_{\mathrm{OH}}$ from 3400 cm^{-1} in pure glass to 3330 cm^{-1} in filled PG and the absence of the OH monomer band at $\nu^{\mathrm{m}}_{\mathrm{OH}}$ 3747 cm^{-1}. This shift $\nu^{\mathrm{ac}}_{\mathrm{OH}}$ to lower frequencies shows that the H-bonds in the latter case are stronger than the H-bonds in pure PG (between part of Si–OH groups and H_2O absorbed in pores).

The estimation of the change of enthalpy ($-\Delta H$) of hydrogen bond when introducing 5CB molecules into the porous glass was made with the use of Ioganson rule [17]:

$$-\Delta H = 0.3(\Delta\nu_{\mathrm{OH}} - 40)^{1/2},$$

$$\Delta\nu_{\mathrm{OH}} = \nu^{\mathrm{m}}_{\mathrm{OH}} - \nu^{\mathrm{ac}}_{\mathrm{OH}}.$$

It was found that, in the case of the filled porous glass, the energy of the hydrogen bond increased by 0.6 kcal mol^{-1}. Note that in the system 5CB–aerosil (ca = 2.5 wt.%) which we studied earlier [8], more weak H-bonds of the type Si–OH···π electrons of the benzene ring formed between Si–OH groups of aerosil and 5CB molecules. In this case, new bands in the region 3650–3550 cm^{-1} appeared which are characteristic for this type of hydrogen bond, and the enthalpy of formation of these H-bonds, $-\Delta H$ was ca. 2–3 kcal mol^{-1}. In the same time, the position and half-width of the Q(C≡N) band remained constant [2,8].

Note that the band 7326 cm^{-1} caused by the overtone 2ν(OH) of the OH group observed in the spectrum of the porous glass also disappeared when 5CB molecules were introduced into the pores in the glass (Fig. 1).

In order to understand which H-bonds form in the case of filled porous glasses, we performed a detailed comparative study of the parameters of IR bands caused by vibrations of benzene ring and cyano group in pure 5CB and 5CB introduced into the porous glass. The maximal shift of the maximum position of the band was observed for the Q(C≡N) band, and was found to be equal to 8 cm^{-1} for higher frequencies (Table 3). As can be seen from the obtained data, the formation of the dimers leads to a shift of the frequency into the low-frequency region and an essential broadening of the band as compared with the case of the 5CB monomer.

As it is known from the data obtained by X-ray diffraction methods, in SC and LC states, the 5CB molecules form dimers due to dipole–dipole interactions between cyanophenyl core [14,15].

When 5CB was introduced into the PG, an additional broadening of the Q(C≡N) band and high-frequency shift of its maximum occurs. In this case, the overtones and combination tones of the stretching and in plane deformation vibrations of the phenyl rings practically remain a constant. This makes it possible to draw a conclusion that the interaction of 5CB molecules with active Si–OH centers of the surface of the porous glass occurs via the formation of hydrogen bonds between Si–OH···N≡C groups. In this case, as pointed before, the enthalpy of H-bond is slightly stronger than between the molecules of PG and water.

As shown by ab initio calculations for molecules with CN groups adsorbed on metal surface, the frequency of Q(C≡N) band of the C≡N group decreases when this group is attached to the surface by the carbon atom or lies parallel to it, and increases when it is attached by nitrogen atom compared with one in gaseous phase [18]. One can suppose that in our case (see Table 3) also the formation of enough strong Si–OH···N≡C–bonds take place.

3.5. IR spectra of LC 5CB in molecular sieves

Unfortunately, in the case of porous glasses, it seems practically impossible to study fundamental characteristic vibrations of phenyl ring and alkyl radical. Such a study was performed for molecular sieve systems of the types MCM and CuMCM filled with 5CB. From Fig. 3 one can see that the position and half-width of the Q(C≡N) band show changes similar to those of the porous glass (Table 3). We have performed graphical separation of the bands in the 2150–2300 cm^{-1} region for the systems under study (Fig. 3(b) and (c)). Two bands were separated in this region. The first band (I) coincides, in its position and half-width, with the band of pure 5CB in NLC phase (dimers), and the second (II), with a maximum at 2236 cm^{-1} and a half-width of about 23 cm^{-1} can be assigned to vibrations Q(C≡N) of the 5CB molecules interacting with active centers of the channels (Al–OH and Si–OH) of the molecular sieves. The intensities S of the bands I and II correspond to the amounts of 5CB molecules in the dipole–dipole bound dimers and 5CB molecules interacting with the surface of the channels, respectively. The amount of the latter decreases when Cu atoms are introduced into the channel. It is in accordance with the fact that Cu atoms can block several hydroxyl groups of the surface. This leads to an increasing amount of 5CB in the LC state (Table 3). From the geometry of the channels, one can suppose that long axes of 5CB dimers are oriented along the axes of the channels in CuMCM. In the case of MCM, some part of 5CB molecules which form hydrogen bonds with active centers are tilted with respect to the axes of the channels and hinder the complete filling of the channels. This was observed from the comparison of the spectra of the MCM and CuMCM filled with 5CB (under the same condition of filling) normalized by the maximum of the ν(Si–O) band with $\nu = 1075\ \mathrm{cm}^{-1}$. It was shown that, in the case of MCM, the intensities of the bands of introduced 5CB is several times lesser.

Consider the stretching and deformational vibrations of the phenyl rings of 5CB in the fundamental vibrations region. As one can see from Fig. 5, the position and half-width of Q(C$\overset{...}{-}$C) vibrations 1604 and 1496 cm^{-1} do not change when introducing 5CB into molecular sieves. Note, however, that the ratio of intensities of these bands (1604 and 1496 cm^{-1}) does change, and can be assigned to different symmetry of these vibrations (A_{1g} and B_{2u}, respectively) in the p-substituted benzene [19]. At the same time, in the region of out-of-plane vibrations, ρ(C$\overset{...}{-}$C–H) 800–850 cm^{-1} and γ(C$\overset{...}{-}$C$\overset{...}{-}$C) 540–570 cm^{-1} of the phenyl ring we observe a redistribution of intensities and changes in positions of the bands (Fig. 4, Table 1).

One can suppose that, when introducing 5CB into the channels or pores with active centers, some part of the dimers dissociate into monomers, which then form enough strong bonds (of H-bond type) with hydroxyl groups of the surface.

In this case, the out-of-plane vibrations of the phenyl rings (polarized perpendicularly to the channel) undergo the most pronounced influence of the surface or volume confinement as compared with Q(C$\overset{...}{-}$C) 1604 and 1496 cm^{-1} vibrations (polarized along the channel).

4. Conclusions

This work shows the following:

1. Nematic liquid crystals can be included into the inner space of the porous glasses and aluminosilicate mesoporous molecular sieves of the MCM-41 type.
2. The liquid crystal molecules interact with the active centers located on the internal surface of the channels, and pores formed enough strong hydrogen bonds of the –C≡N···O–H–Al(Si) type. It was proved by the appearance of a new IR absorption band in the Q(C≡N) stretching vibration region, by the decrease in the number of the trapped molecules when the active centers are partially blocked by the Cu^{2+} cations. In case of LC–Aerosil system, only weak H-bonds were formed between Si–OH···π electrons of the benzene ring.
3. It was shown that some 5CB molecules exist in the dimer state which are characteristic of the liquid crystal phase.

Acknowledgements

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