

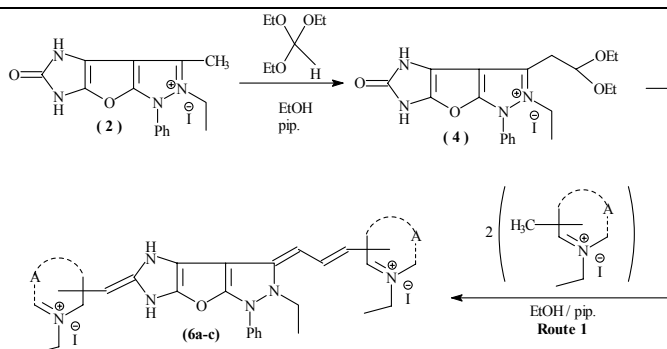
POLYHETEROCYCLIC COMPOUND IN THE SYNTHESIS AND SPECTRAL STUDIES OF SOME NOVEL METHINE CYANINE DYES

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New polyheterocyclic compound having the nucleus of furo[(3,2-d); pyrazolinium; (3,2-d) imidazole] iodide salt were prepared and employed to synthesis of different classes of some novel methine cyanine dyes. The synthesized cyanine dyes cover monomethine cyanine dyes, trimethine cyanine dyes, and mono/tri-mixed methine cyanine dyes. The electronic visible absorption spectral of all the synthesized cyanine dyes were examined in 95% ethanol solution. Structural determinations were carried out through elemental analysis, visible spectra, mass spectrometer, IR and ^1H NMR spectroscopic data.



INTRODUCTION

Cyanine dyes¹⁻¹⁴ are interesting class of organic heterocyclic dyes which have a wide spectrum of uses and applications in several fields of science and technology. These include but are not limited to spectral and/or photographic sensitizers in manufacturing technology of photographic materials industry, as optical recording materials in laser disks, as sensitizers in solar cells, as photodynamic therapy (PDT) tools, as fluorescent labels and probes for cells, micells and organelles. In addition, nanofilms of cyanine dyes¹⁵⁻²⁸ are widely used in recording media of write-once compact disks, in photovoltaic, in photosynthetic systems, and in medicine as components of medical microprobes and microcapsules.

Taking in account the above benefits of cyanine dyes, in this research paper we prepared new

photosensitizer, solvatochromic and halochromic cyanine dyes as new synthesis contribution and spectroscopic investigation in this field to be used and/or applied as photographic sensitizer in photosensitive material industry, as probes for determining solvent polarity in physical, physical organic and/or inorganic chemistry and/or as acid-base indicators in analytical chemistry.

RESULTS AND DISCUSSION

1. Synthesis

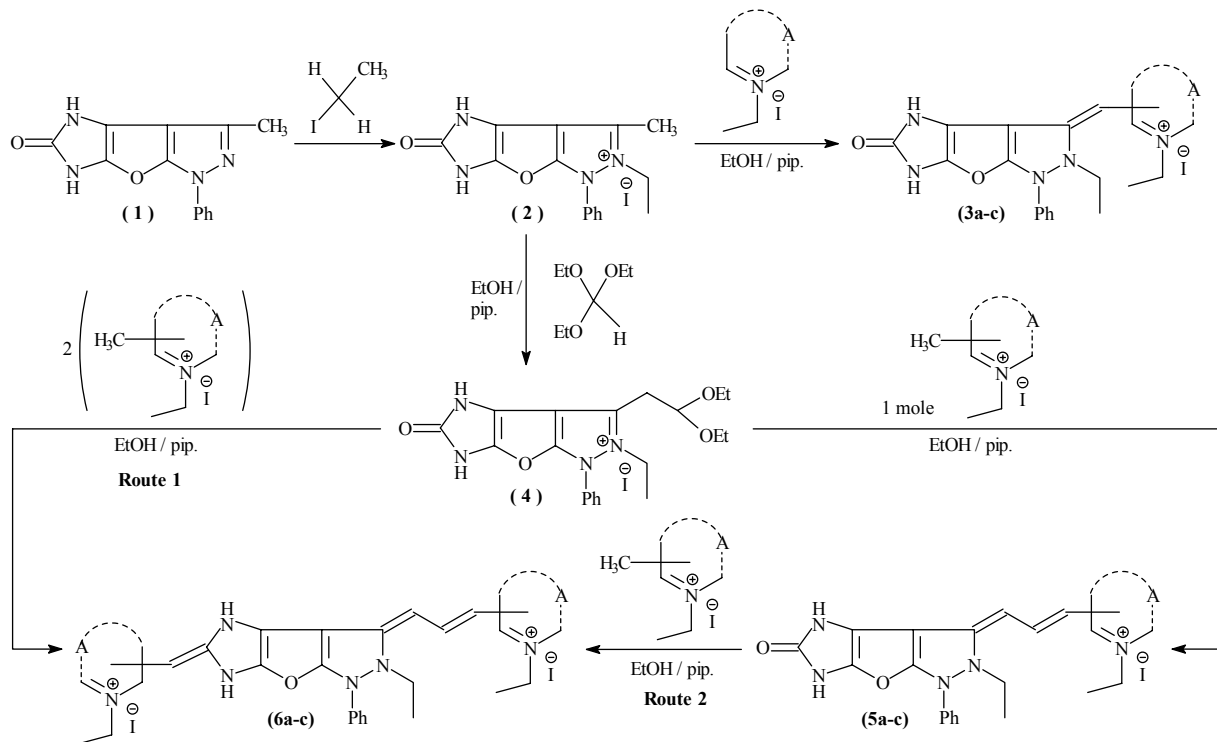
Quaternization of 4-methyl-6-oxo-2-phenyl-furo[(3,2-d)]pyrazole; (3,2-d) imidazole] (1) using excess of iodoethane resulted in 3-iodoethane quaternary salt (2), Scheme (1), Table (1).

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Reaction of (2) and iodoethane quaternary salts of pyridine, quinoline and/or isoquinoline in ethanol as organic solvent and piperidine as a basic catalyst gives the 4[4(1)]-monomethine cyanine dyes (3a-c), Scheme (1), Table (1).

From the reaction of equimolar ratios of (2) and triethylorthoformate in ethanol catalyzed by piperidine resulted the intermediate compound (4),

Scheme (1), Table (2). Further reaction of the intermediate compound (4) with N-ethyl(α -picolinium, quinaldinium and/or γ -picolinium) iodide quaternary salts in equimolar ratios in ethanol containing few mls of piperidine achieved the 4[2(4)]-trimethine cyanine dyes (5a-c), Scheme (1), Table (2).

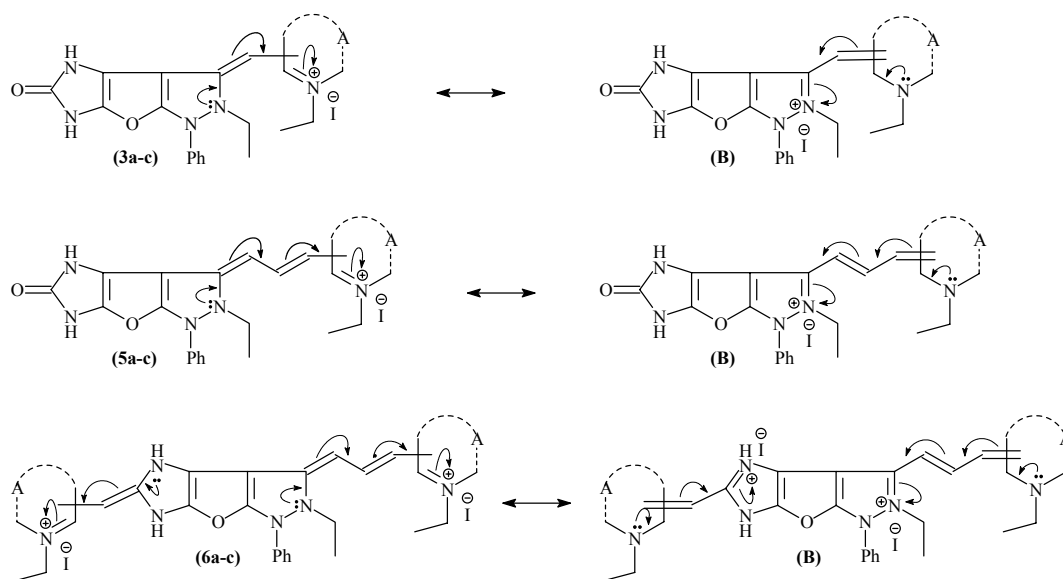


Scheme (1)

Substituents in Scheme 1:

(3a-c): A=1-ethyl-pyridinium-4-yl salt (a); 1-ethyl-quinolinium-4-yl salt (b); 2-ethyl isoquinolinium-1-yl salt (c).

(5a-c); (6a-c): A=1-ethyl=pyridinium-2-yl salt (a); 1-ethyl-quinolinium-2-yl salt (b); 1-ethyl pyridinium-4-yl salt (c).



Scheme 2

Table 1

Characterization of the prepared compounds (2) and (3a-c)

Comp. No.	Nature of products			Molecular formula (M. Wt.)	Analysis %						Absorption spectra in 95% ethanol solution	
	Colour	Yield (%)	M.p. (°C)		Calculated			Found			λ_{\max} (nm)	ϵ_{\max} (mole ⁻¹ cm ⁻²)
					C	H	N	C	H	N		
2	Deep brown	61	180	C ₁₅ H ₁₅ N ₄ O ₂ I (410)	43.90	3.65	13.65	43.85	3.52	13.57	-----	-----
3a	Red	41	177	C ₂₂ H ₂₂ N ₅ O ₂ I (515)	51.26	4.27	13.59	51.11	4.20	13.43	440	4600
3b	Violet	68	170	C ₂₆ H ₂₄ N ₅ O ₂ I (565)	55.22	4.24	12.38	55.21	4.19	12.28	451	21700
3c	Deep red	55	190	C ₂₆ H ₂₄ N ₅ O ₂ I (565)	55.22	4.24	12.38	55.18	4.15	12.16	445	11000

Table 2

Characterization of the prepared compounds (4), (5a-c) and (6a-c)

Comp. No.	Nature of products			Molecular formula (M. Wt.)	Analysis %						Absorption spectra in 95% ethanol solution	
	Colour	Yield (%)	M.p. (°C)		Calculated			Found			λ_{\max} (nm)	ϵ_{\max} (mole ⁻¹ cm ⁻²)
					C	H	N	C	H	N		
4	Brown	61	130	C ₂₀ H ₂₅ N ₄ O ₄ I (512)	46.87	4.88	10.93	46.74	4.77	10.89	-----	-----
5a	violet	50	120	C ₂₄ H ₂₃ N ₅ O ₂ I (540)	53.33	4.25	12.96	53.10	4.21	12.92	380, 530	23000, 11000
5b	Deep violet	65	145	C ₂₈ H ₂₅ N ₅ O ₂ I (590)	56.94	4.23	11.86	56.89	4.20	11.77	387, 540	43000, 11000
5c	Violet	45	100	C ₂₄ H ₂₃ N ₅ O ₂ I (540)	53.33	4.25	12.96	53.19	4.17	12.86	383, 535	20000, 14000
6a	Violet	52	125	C ₃₂ H ₃₃ N ₆ OI ₂ (771)	49.80	4.28	10.89	49.76	4.18	10.76	385, 535	14000, 12000
6b	Deep violet	60	135	C ₄₀ H ₃₇ N ₆ OI ₂ (871)	55.10	4.24	9.64	55.00	4.17	9.53	390, 545	15000, 180000
6c	Deep violet	45	135	C ₃₂ H ₃₃ N ₆ OI ₂ (771)	49.80	4.28	10.89	49.70	4.20	10.80	385, 540	16000, 80000

Table 3

IR and ¹H NMR (mass) spectral data of the prepared compounds

Comp. No	IR Spectrum (KBr, Cm ⁻¹)	¹ H NMR Spectrum (DMSO, δ); & (Mass data)
2	646, 687 (monosubstituted phenyl). 1074, 1119, 1157 (C-O-C cyclic). 1548, 1454, 1493 (C=N). 1595 (C=C). 1716 (C=O). 2922, 2854 (quaternary salt). 3415 (NH).	2.15 (s, 3H, CH ₃ of position 4). 2.25-2.30 (m, 3H, CH ₃ of position 3). 3.9 (b, 2H, CH ₂ of position 3). 7.2 (m, 2H, 2NH) 7.4-8 (m, 5H, aromatic). M ⁺ : 410
3b	647, 694 (monosubstituted phenyl). 755, 814 (o. disubstituted phenyl). 1119, 1164 (C-O-C) cyclic) 1493, 1450 (C=N). 1599 (C=C). 1712 (C=O). 2921, 2854 (quaternary salt). 3421 (NH).	0.8-1 (b, 3H, CH ₃ of N-quinolinium). 1.1-1.4 (b, 2H, CH ₂ of N-quinolinium). 2.2 (m, 3H, CH ₃ of position 3). 3.3 (m, 2H, CH ₂ of position 3). 7.0 (b, 2H, 2NH). 7.2-8.4 (m, 12H, aromatic + heterocyclic + -CH=).
4	648, 692 (monosubstituted phenyl). 1027, 1067, 1122, 1160 (C-O-C cyclic). 1496, 1450 (C=N). 1596 (C=C). 1718 (C=O). 2924, 2853 (quaternary salt). 3421 (NH)	0.8-0.9 (b, 6H, 2CH ₃ of diethoxy ethyl). 1.3 (d, 2H, CH ₂ of position 4). 1.6 (b, 4H, 2CH ₂ of diethoxy ethyl). 2.4 (m, 1H, -CH- of position 4). 3.0 (b, 3H, CH ₃ of position 3). 3.4 (b, 2H, CH ₂ of position 3). 7.15 (b, 2H, 2NH). 7.2-8.2 (m, 5H, aromatic). M ⁺ : 512
5b	626, 692 (monosubstituted phenyl). 755 (o. disubstituted phenyl). 1026, 1086, 1159 (C-O-C cyclic). 1494, 1451 (C=N). 1624, 1596 (C=C). 1716 (C=O). 2925, 2855 (quaternary salt). 3431 (NH).	0.8-1 (b, 3H, CH ₃ of N-quinolinium). 1.2-1.6 (b, 2H, CH ₂ of N-quinolinium). 2.3 (m, 3H, CH ₃ of position 3). 3.3 (m, 2H, CH ₂ of position 3). 7.1 (b, 2H, 2NH). 7.2-8.2 (m, 14H, aromatic + heterocyclic + 3 -CH=).
6b	647, 693 (monosubstituted phenyl). 755, 825 (o. disubstituted phenyl). 1032, 1085, 1130, 1158 (C-O-C cyclic). 1494, 1449 (C=N). 1596 (C=C). 2925, 2856 (quaternary salt). 3428 (NH).	0.8 (b, 3H, CH ₃ of N-quinolinium of position 4). 1.2 (b, 2H, CH ₂ of N-quinolinium of position 4). 1.6 (b, 3H, CH ₃ of N-quinolinium of position 6). 2.1 (b, 2H, CH ₂ of N-quinolinium of position 6). 2.3 (m, 3H, CH ₃ of position 3). 3.3 (m, 2H, CH ₂ of position 3). 7.1 (b, 2H, 2NH). 7.2-8.4 (m, 21H, aromatic + heterocyclic + 4 -CH=).

1:2 molar reaction of the intermediate compound (4) and ethyl iodide quaternary salts of α -picoline, quinaldine and/or γ -picoline in ethanol and presence of piperidine achieved the 6[2(4)]-mono-4[2(4)]-tri-mixed methine cyanine dyes (6a-c), Scheme (1), Route (1), Table (2).

Chemical confirmation takes place for the mixed methine cyanine dyes (6a-c) via route (2) by reaction of the previously prepared trimethine cyanine dyes (5a-c) with iodoethane quaternary salts of α -picoline, quinaldine and/or γ -picoline in equimolar ratios, in ethanol as organic solvent and piperidine as a basic catalyst to give the same mixed methine cyanine dyes (6a-c) obtained by

route (1), characterized by melting points, mixed melting points, same IR and ¹H NMR spectral data, Scheme (1), Route (2), Table (2).

The structure of the prepared compounds was confirmed by elemental analysis, Tables (1) and (2), visible spectra, Tables (1) and (2), mass spectrometer, Table (3), IR²⁹ and ¹H NMR³⁰ spectroscopic data, Table (3).

2. Spectral studies in 95% ethanol solution

The electronic visible absorption spectra of the monomethine cyanine dyes (3a-c) in 95% ethanol solution disclose bands in the visible range 440-

451 nm. The positions of these bands and their molar extinction coefficients are influenced by the nature of the heterocyclic quaternary salts residue and their linkage positions. So, substituting A=1-ethyl-pyridinium-4-yl salt by A=1-ethyl-quinolinium-4-yl salt, transferring from dye (3a) to dye (3b) makes bathochromic shifts for the absorption bands by 11 nm accompanied by increasing the intensity of the absorption band, Scheme (1), Table (1). This may be attributed to increasing π -delocalization conjugation in dye (3b) due to the presence of quinoline ring system in correspondence to pyridine ring system in dye (3a). Changing the linkage positions from A=1-ethyl-quinolinium-4-yl salt in dye (3b) to A=2-ethyl-isoquinolinium-1-yl salt in dye (3c) resulted in a hypsochromic shift by 6 nm in addition to decreasing the intensity of the absorption band, Scheme (1), Table (1). This can be related to decreasing the length of π -delocalization conjugation in latter dye (3c) due to the presence of isoquinoline ring system in correspondence to quinoline ring system in former dye (3b).

Additionally, the electronic visible absorption spectra of trimethine cyanine dyes (5a-c) in 95% ethanol solution discloses bands in the visible region 380-540 nm. The positions of these bands underwent displacements to give bathochromic and/or hypsochromic shifts accompanied by increasing and/or decreasing the intensity of the bands depending upon the nature of the heterocyclic quaternary residue (A) and their linkage positions. So, substituting α -picoline quaternary salt residue in dye (5a) by quinaldine quaternary salt residue to obtain dye (5b) causes bathochromic shifts by 10 nm in addition to increasing the intensity of the absorption bands, Scheme (1), Table (2). This can be related to increasing conjugation in the latter dye (5b) due to the presence of quinaldine nucleus in correspondence to α -picoline nucleus in the former dye (5a). Changing the linkage positions from 2-yl salt residue in α -picoline dye (5a) to 4-yl salt residue in γ -picoline dye (5c) resulted in a red shift for the absorption bands by 5 nm accompanied by increasing the intensity of the absorption bands, Scheme (1), Table (2). This can be attributed to increasing the length of the π -delocalization conjugation in latter dye (5c) due to the presence of γ -picoline ring system in correspondence to α -picoline ring system in the former dye (5a).

Also, the electronic visible absorption spectra of the mono/tri- mixed methine cyanine dyes (6a-c) gives bands in the visible region 385-545 nm. The positions of these bands and their molar extinction coefficients are remarkably effected by the nature of the heterocyclic quaternary salt residue (A) and their linkage positions, Scheme (1), Table (2). This can be owed to the same reasons mentioned before in the case of the trimethine cyanine dyes (5a-c).

The comparison of the electronic visible absorption spectra of the monomethine cyanine dyes (4a-c) with those of the trimethine cyanine dyes (6a-c) reveals that the latter dyes have bathochromic shifted and intensified bands than the former dyes, in addition to an increase in number of the absorption bands. This can be related to increasing the number of methine groups between the basic center (nitrogen atom) and the acidic center (quaternary salt) in latter dyes, Scheme (1), Tables (1) and (2).

Comparing the electronic visible absorption spectra of the mono/tri- mixed methine cyanine dyes (6a-c) with those of the monomethine cyanine dyes (3a-c) and/or the trimethine cyanine dyes (5a-c) showed that the former mono/tri- mixed methine dyes (6a-c) have red shifted and intensified absorption bands in comparison to the latter monomethine cyanine dyes (3a-c) and the trimethine cyanine dyes (5a-c), Scheme (1), Tables (1), (2). This can be attributed to two factors. The first factor is related to increased number of methine units in the former mixed methine cyanine dyes (6a-c) in comparison to the latter monomethine cyanine dyes (3a-c) and the trimethine cyanine dyes (5a-c). The second factor is related to presence of two electronic charge transfer pathways inside the mono/tri- mixed methine cyanine dyes molecules (6a-c) in correspondence to one electronic charge transfer pathways inside the molecules of the monomethine cyanine dyes (2a-c) and the trimethine cyanine dyes (5a-c), Scheme (2).

General comparison of the electronic absorption spectra bands of the monomethine cyanine dyes (3a-c), trimethine cyanine dyes (5a-c) and/or the mono/tri- mixed methine cyanine dyes (6a-c) reveals that the bathochromic shifts and/or the red shifts of the bands positions increase by increasing the number of the methine units in these dyes in the order of: mono/tri- mixed methine cyanine dyes > trimethine cyanine dyes > monomethine cyanine dyes.

EXPERIMENTAL

1. General

All the melting points of the prepared compounds are measured using Electrothermal 15V, 45W 1 A9100 melting point apparatus, Chemistry department, Faculty of Science (Aswan University) and are uncorrected. Elemental analysis were carried out at the Microanalytical Center of Cairo University by an automatic analyzer (Vario EL III Germany). Infrared spectra were measured with a FT/IR (4100 Jasco Japan), Cairo University. ¹H NMR Spectra were accomplished using Varian Gemini-300 MHz NMR Spectrometer (Cairo University). Mass Spectroscopy were recorded on Mas 1: GC-2010 Shimadzu Spectrometer (Cairo University). Electronic visible absorption spectra were carried out on Visible Spectrophotometer, Spectro 24 RS Labomed, INC, Chemistry department, Faculty of Science (Aswan University).

2. Synthesis

2.1. Synthesis of 3-ethyl-4-methyl-6-oxo-2-phenyl-furo

[(3,2-d) pyrazolium, (3,2-d) imidazole] iodide salt (2)

A pure crystallized sample of (1) (0.01 mol) and equimolar ratio of iodoethane were heated in a sealed tube till complete fusion. The products were cooled, dissolved in ethanol (30 mL) and heated under reflux for 1 hr. The reaction mixture were filtered while hot to remove impurities, concentrated and cooled. The precipitates were collected, dried, and crystallized using ethanol. See data in Table 1.

2.2. Synthesis of 3-ethyl-6-oxo-2-phenyl-furo [(3,2-d)

pyrazole, (3,2-d) imidazole]-4[4(1)]-monomethine cyanine dyes (3a-c)

Equimolar ratios (0.01 mol) of the quaternized compound (2) and N-ethyl (pyridinium, quinolinium or isoquinolinium) iodide salts were dissolved in ethanol (30 mL) containing piperidine (1-2 mL). The reaction mixture were heated under reflux for 6 hrs, It was filtered off while hot to remove any impurities, cooled, and precipitated by dilution with ice-water mixture with continues shaking. The precipitates were filtered, washed with water several times, dried and crystallized from ethanol. Relevant data are given in Table 1.

2.3. Synthesis of 3-ethyl-6-oxo-2-phenyl-furo [(3,2-d)

pyrazole, (3,2-d) imidazole]-4(1,1-diethoxy ethyl) iodide salt (4) as intermediate compound (4)

A mixture of equimolar ratios (0.01 mol) of the quaternary salt (2) and triethylorthoformate were dissolved in ethanol (30 ml) containing piperidine (1-2 mL) and heated under reflux for 6 hrs, until it gave a permanent deep colour at the end of refluxing. It was filtered while hot to remove impurities, concentrated to one half its volume, cooled and precipitated by cold water. The precipitates were filtered, washed with water, dried and crystallized from ethanol. The data were listed in Table 2.

2.4. Synthesis of 3-ethyl-6-oxo-2-phenyl-furo [(3,2-d)

pyrazole, (3,2-d) imidazole]-4[2(4)]-trimethine cyanine dyes (5a-c)

Equimolar ratios (0.01 mol) of the intermediate compound (4) and N-ethyl (2-picolinium, quinaldinium or 4-picolinium) iodide salts were dissolved in ethanol (30 mL)

containing piperidine (1-2 mL). The reaction mixture was heated under reflux for 8 hrs, and attained a highly violet colours at the end of refluxing. The mixture was filtered off while hot, precipitated by dilution with ice-water mixture with continues shaking. The precipitates were filtered off, washed with water several times, dried and crystallized from ethanol. The data were registered in Table 2.

2.5. Synthesis of 3-ethyl-2-phenyl-furo [(3,2-d) pyrazole,

(3,2-d) imidazole]-4[(2(4)]tri- 6[2(4)]mono- mixed methine cyanine dyes (6a-c)

This synthesis was accomplished via two different routes.

Route 1: Unimolar ratios of intermediate compound (4) (0.01 mol) and bimolar ratios (0.02 mol) of N-ethyl(2-picolinium, quinaldinium or 4-picolinium) iodide salts were dissolved in ethanol (30 mL) containing piperidine (1-2 mL) and heated under reflux for 3-5 hrs, and attained a highly violet colours at the end of refluxing. The mixture was filtered off while hot, cooled and precipitated by dilution with ice-water mixture with continues shaking. The precipitates were filtered, washed with water, dried and crystallized from ethanol. The data were recorded in Table 2.

Route 2: Piperidine (1-2 mL) was added to a mixture of equimolar ratios (0.01 mol) of the previously prepared compounds (5a-c) and iodoethane quaternary salts of α -picoline, quinaldine or γ -picoline dissolved in ethanol (30 mL). The reaction mixture was heated under reflux for about 3-5 hrs until attained permanent violet colour at the end of refluxing. It was filtered off while hot, concentrated, and cooled. The precipitated products, which appeared on dilution with cold water were filtered off, washed several times with water, dried and crystallized from ethanol to give the same 4[2(4)]tri- 6[2(4)]mono- mixed methine cyanine dyes (6a-c) obtained by route 1, characterized by the same melting points, mixing melting points, same IR and ¹H NMR spectral data. The data were summarized in Table 2.

3. Spectral studies

The electronic visible absorption spectra of the prepared cyanine dyes were examined in 95% ethanol solution and recorded using 1 cm Q_z cell in Visible Spectrophotometer, Spectro 24 RS Labomed, INC. A stock solution (1 x 10⁻³M) of the dyes was prepared and diluted to a suitable volume in order to obtain the desired lower concentrations. The spectra were recorded immediately to eliminate as much as possible the effect of time.

CONCLUSIONS

From the above discussed results we can conclude that:

The electronic visible absorption spectra of the synthesized cyanine dyes in 95% ethanol solution underwent displacements to give bathochromic and/or hypsochromic shifted bands depending upon the following factors:

Types of the heterocyclic quaternary salts residue in the order of: quinoline dyes > pyridine dyes.

Linkage position of the heterocyclic quaternary salts residue in the order of quinoline dyes > isoquinoline dyes, 4-picoline dyes > 2-picoline dyes.

Number of methine units in the dyes molecule in the order of: mixed methine cyanine dyes > trimethine cyanine dyes > monomethine cyanine dyes.

The number of the electronic charge transfer pathways inside the dyes molecules in the order of: two electronic charge transfer pathways > one electronic charge transfer pathway.

The intensity of the colours of the synthesized cyanine dyes can be attributed to two suggested mesomeric structures (A) and (B) producing a delocalized positive charge over the conjugated system, Scheme (2).

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