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Synthesis, Vibrational spectroscopic investigation, and structure study of some bismaleimide

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Abstract

The N,N'-alkyl and aryl bismaleimide (BMIs) have been synthesized and studied spectroscopically and X-ray single crystal analysis. The structure of bismaleimides was confirmed by Elemental analyses, EI-mass, FT-IR, ¹HNMR, ¹³CNMR, DEPT-135 NMR, COSY, HSQC and HMBC spectral data and X-ray of BMI7. Infrared spectra of serial from bismaleimides have experimentally been reported in the region of 4000–600 cm⁻¹ to study the effect of spacer between two maleimides moieties, by three type of spacer, first direct N-N bonded, second with different aliphatic serial and finally by two different aromatic molecular as spacer. The position of the absorption bands for some functional groups (C=O, C=C, C-H and C-N) varies depending on the type and length of the link spacer (R) between the two maleimide rings moieties on the studied bismaleimides. The optimized molecular structure has been investigated experimentally and theoretically using Gaussian09 software package. DFT calculations were carried out by using B3LYP functional with 6-311G(d,p) basis set.

Key words: bismaleimide BMIs, Vibrational spectroscopic investigation, X-ray, DFT

1 Introduction

Bismaleimide (BMI) resins as a kind of high-performance materials [1], are used in a wide range of applications from commodity materials to hi-tech aerospace industries, such as multiplayer printed circuit boards for large scale computers [2,3]. These resins have high thermostability, tensile strength and modulus, good flame resistance, excellent chemical and corrosive resistance, as well as low water sensitivity [4]. The bismaleimide resins have attracted much attention in the fields of advanced composites due to their excellent processing

characteristics without the formation of volatile by-products and outstanding thermomechanical and flammability behavior on the finally cured state [5]. Bismaleimide possesses some remarkable properties such as low moisture absorption, highly crosslinked structures, high chemical resistance, high mechanical stability, etc. These advantages make bismaleimide applied widely in adhesive, packaging and aerospace industry [6].

Bismaleimides (BMIs) are versatile building blocks in polymer synthesis. BMIs are used for the synthesis of high molecular

weight, step-growth polymers, and serve as effective cross-linkers. BMI thermosetting addition polyimide resins are the key components of aeroengines and aircraft because of their high temperature stability, hot-wet strength, and fatigue resistance [7]. BMI crosslinkers are commonly used to explore and characterize protein structure or interactions by site specific biomolecule modification (i.e. click chemistry), and forming crosslinks between sites in a protein oligomer or interaction can assist in determining intra- and intermolecular distances. N-Alkyl bismaleimides find application as water soluble thiol-reactive

2- Experimental

2-1 Materials and methods

All chemicals purchased were of reagent grade and used without further purification. Determinations of infrared spectra were performed in KBr disc using FTIR spectrophotometer Model Bruker Tensor . Melting point was determined by

2-2 General Procedure for the synthesis of bismaleimide (BMI1-BMI7)

Desired bismaleimides precursors were prepared according to the classical approach for the synthesis of N-alkyl (or N-aryl) BMIs follows Searle's method. A scheme of mechanism steps of dehydration reaction of bismaleimide BMI1-BMI7 is illustrated in scheme 1.

Maleic anhydride (0.042mol) in 25 mL diethyl ether (acetone for compounds BMI6 and BMI7) was reacted with, diamine (hydrazine for compound BMI1) (0.02mol) in 50 mL diethyl ether (acetone for compounds BMI6 and BMI7) to give the corresponding bismaleamic acid. In addition, the mixture was stirred for 30 min at 0°C, then the temperature was raised to 35°C. Over a period of 1h. Bismaleamic

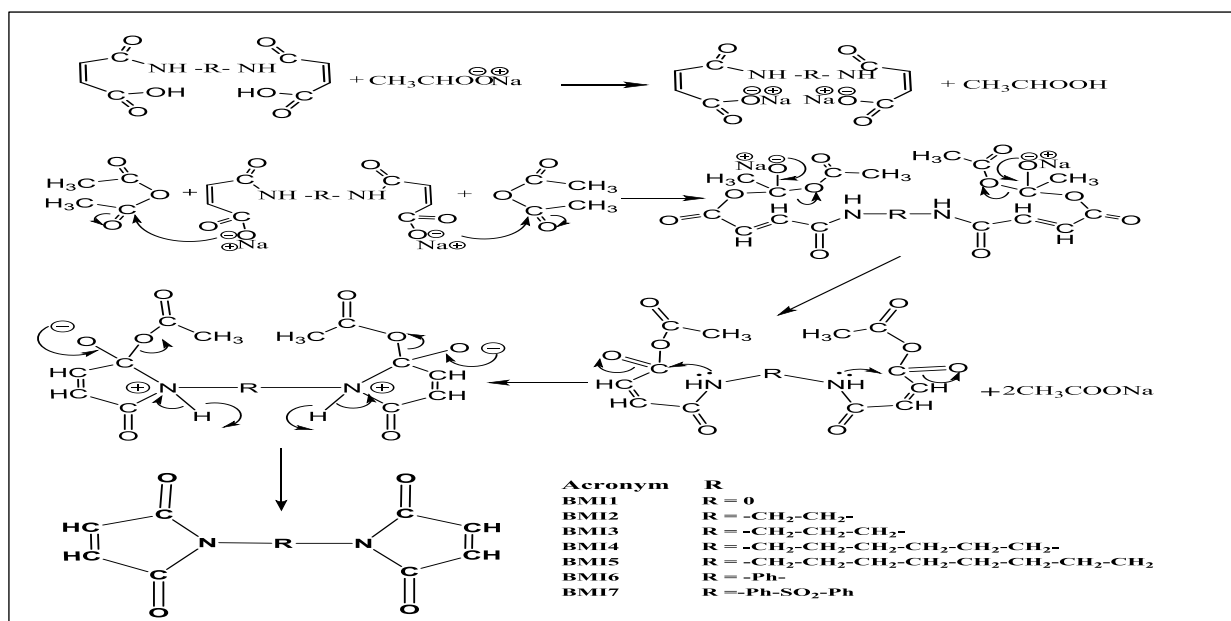
homobifunctional cross-linkers. BMIs have found application in the production of multimeric forms of the T cell surface glycoprotein CD4 through a sugar-based crosslinking strategy, synthesis of bradykinin (BK) antagonist dimers, and crosslinking of diene-modified DNA [8].

We required N-alkyl BMIs in gram quantities. Although commercially available, the high cost of commercial BMIs has driven us to look for an efficient and economic synthesis of these compounds in our laboratory and on the other hand we had a detailed spectral for this class of compounds study.

Buechi 530 melting point. 1D and 2D-NMR spectra were recorded on a Bruker NMR spectrometer Avance DRX 400, using DMSO-d⁶ as solvent and tetramethylsilane as an internal standard.

acid (0.024 mol) was refluxed in the mixture of (0.176 mol) acetic anhydride, (0.043 mol) sodium acetate for 3 hr., then the solution was cooled down to room temperature and poured into ice-cold water under stirring and left to be sedimented. Then the solid substance obtained was filtered. The product was washed with a solution of sodium carbonate (10%) until it became free from acetic acid. Finally, it was washed with water and dried in vacuum at a temperature of 80°C.

Structure of bismaleimides (BMI1-BMI7) was confirmed by Elemental analyses, EI-mass, FT-IR, ¹HNMR, ¹³CNMR, DEPT-135 NMR, COSY, HSQC and HMBC spectral data and X-ray for BMI7.



Scheme 1. Mechanism steps of dehydration reaction of bismaleimide BMI1-BMI7.

¹H and ¹³C NMR spectra of bismaleimides (BMI1-BMI7) as DMSO solutions were recorded at 400 MHz, at 298 K. Chemical shifts (δ) are quoted in ppm and referenced to internal TMS. Data are reported as follows: s, singlet; d, doublet; t, triplet; m, multiplet; 2D NMR techniques (COSY, HSQC and HMBC) were used to assist in structure elucidation. IR spectra were reported in reciprocal centimeters (cm⁻¹). Accurate mass were determined by and electronic impact (EI).

[1,1'-bipyrrole]-2,2',5,5'-tetraone BMI1

White crystals, m.p. 165-167°C. Anal. Calcd. C₈H₄N₂O₄: C, 50.01; H, 2.10; N, 14.13. Found: C, 49.01; H, 2.27; N, 14.58. IR (KBr, cm⁻¹) 3101, 1728, 1576, 1368; ¹H NMR (DMSO-*d*⁶) δ 7.408 (s 4H, 2-CH=CH); ¹³C NMR δ 166 (C=O), 135 (CH=CH); DEPT-135, up signal 135; HSQC (6.31, 132.8), (6.42, 127.29); HMBC (6.42, 165.65), (6.39, 162.06), (6.42, 127.29), (6.39, 132.4); (m/z) [M⁺] = calcd for C₈H₄N₂O₄ 192.0, found 192.0.

1,1'-(ethane-1,2-diyl)bis(1H-pyrrole-2,5-dione) BMI2.

Brown crystals, m.p. 186°C. Anal. Calcd. C₁₀H₈N₂O₄: C, 54.55; H, 3.66; N, 12.72. Found: C, 53.94; H, 3.78; N, 12.42. IR (KBr, cm⁻¹) 3105, 2971, 1687, 1584, 1381; ¹H NMR (DMSO-*d*⁶) δ 7.024 (s 4H, 2-CH=CH), 3.55 (s, 4H, 2CH₂); ¹³C NMR δ

171 (C=O), 135 (CH=CH), 35 (CH₂CH₂); DEPT-135, up signal 135, down signal 35 (CH₂CH₂); HSQC (3.55, 35), (7.024, 135); HMBC (3.55, 35), (3.55, 171); (m/z) [M⁺] = calcd for C₁₀H₈N₂O₄ 220.1, found 220.1.

1,1'-(propane-1,3-diyl)bis(1H-pyrrole-2,5-dione) BMI3.

Brown crystals, m.p. 169°C. Anal. Calcd. C₁₁H₁₀N₂O₄: C, 58.06; H, 4.87; N, 11.29. Found: C, 56.39; H, 4.37; N, 11.48. IR (KBr, cm⁻¹) 3088, 2971, 1687, 1584, 1381; ¹H NMR (DMSO-*d*⁶) δ 7.025 (s 4H, 2CH=CH), 3.26 (t, 2H, 2CH₂), 1.79 (m, 2H, CH₂); ¹³C NMR δ 171 (C=O), 135

(CH=CH), 35 (2CH₂), 27 (CH₂); DEPT-135, up signal 135, down signal 35 (2CH₂), 27 (CH₂); COSY (3.26, 1.79), HSQC (7.025, 135), (3.26, 35), HMBC (3.26, 171), (1.79, 35); (m/z) [M⁺] = calcd for C₁₁H₁₀N₂O₄ 234.1, found 234.1.

1,1'-(hexane-1,6-diyl)bis(1H-pyrrole-2,5-dione) BMI4

White crystals, m.p. 138-140°C. Anal. Calcd. C₁₄H₁₆N₂O₄: C, 60.86; H, 5.84; N, 10.14. Found: C, 59.23; H, 5.76; N, 10.43. IR (KBr, cm⁻¹) 3089, 2940, 1687, 1608, 1373; ¹H NMR (DMSO-*d*⁶) δ 7.01 (s 4H, 2CH=CH), 3.13(t, 4H, 2CH₂), 1.45(m, 4H, CH₂), 1.22(m, 4H, CH₂); ¹³C NMR δ 171

(C=O), 135 (CH=CH), 38 (2CH₂), 27 (2CH₂), 25 (2CH₂) ; DEPT-135, up signal 135, down signal 38(2CH₂), 27 (CH₂), 25 (CH₂) ; COSY (3.13, 1.45), HSQC (3.13,38), (7.01,135); HMBC (7.01,171), (1.22, 171); (m/z) [M⁺] = calcd for C₁₄H₁₆N₂O₄ 276.1, found 276.1.

(1,1'-(octane-1,8-diyl)bis(1H-pyrrole-2,5-dione) BMI5

White crystals, m.p. 127-130°C. Anal. Calcd. C₁₆H₂₀N₂O₄: C, 63.14; H, 6.62; N, 9.20. Found: C, 63.11; H, 6.49; N, 9.14. IR (KBr, cm⁻¹) 3108, 2937, 1689, 1610, 1371; ¹H NMR (DMSO-*d*⁶) δ 7.00 (s 4H, 2CH=CH), 3.37(t, 4H, 2CH₂), 1.46(m, 4H, CH₂), 1.20(m, 4H, CH₂); ¹³C NMR δ 171 (C=O), 135 (CH=CH), 37 (2CH₂), 28.5

(2CH₂), 28 (2CH₂), 26 (2CH₂) ; DEPT-135, up signal 135 down signal 37 (2CH₂), 28.5 (CH₂), 28 (CH₂), 26 (2CH₂); COSY (3.37,1.46), HSQC (1.46,28), (3.37, 37), (7.00, 135); HMBC (7.00,171), (3.37, 171); (m/z) [M⁺] = calcd for C₁₆H₂₀N₂O₄ 304.1, found 304.1.

1,1'-(1,4-phenylene)bis(1H-pyrrole-2,5-dione) BMI6

Pale yellow crystals, m.p. >300°C. Anal. Calcd. C₁₄H₈N₂O₄: C, 62.69; H, 3.01; N, 10.44. Found: C, 61.93; H, 3.22; N, 10.46. IR (KBr, cm⁻¹) 3107, 1696, 1600, 1389; ¹H NMR (DMSO-*d*₆) δ, 7.24 (s 4H, 2CH=CH), 7.46 (s, 4H, 4CH aromatic); ¹³C

NMR δ 170 (C=O), 135 (CH=CH), 131 (C-N aromatic), 128(4CH aromatic); DEPT-135, up signal 135, 128; HMBC (7.24,170), (7.46, 128); (m/z) [M⁺] = calcd for C₁₄H₈N₂O₄ 268.0, found 268.0.

1,1'-(sulfonylbis(4,1-phenylene) bis(1H-pyrrole-2,5-dione) BMI7

Colorless crystals, m.p.245°C. Anal. Calcd. C₂₀H₁₂N₂O₆S: C, 58.82; H, 2.96; N, 6.86. Found: C, 58.50; H, 3.18; N, 6.88. IR (KBr, cm⁻¹) 3106, 1708, 1590, 1392; ¹H NMR (DMSO-*d*₆) δ 8.125 (dd, 4H, 4CH aromatic), 7.643(dd, 4H, 4CH aromatic), 7.24 (s, 4H, 2CH=CH); ¹³C NMR δ 170

(C=O), 139 (N-C aromatic), 137 (S-C aromatic)135 (CH=CH), 128.00(CH aromatic), 127 (CH aromatic); DEPT-135, up signal 135, 128, 127; HSQC (8.125,128), (7.64,127), (7.24,135); HMBC (7.24,170); (m/z) [M⁺] = calcd for C₂₀H₁₂N₂O₆S 408.0, found 408.0.

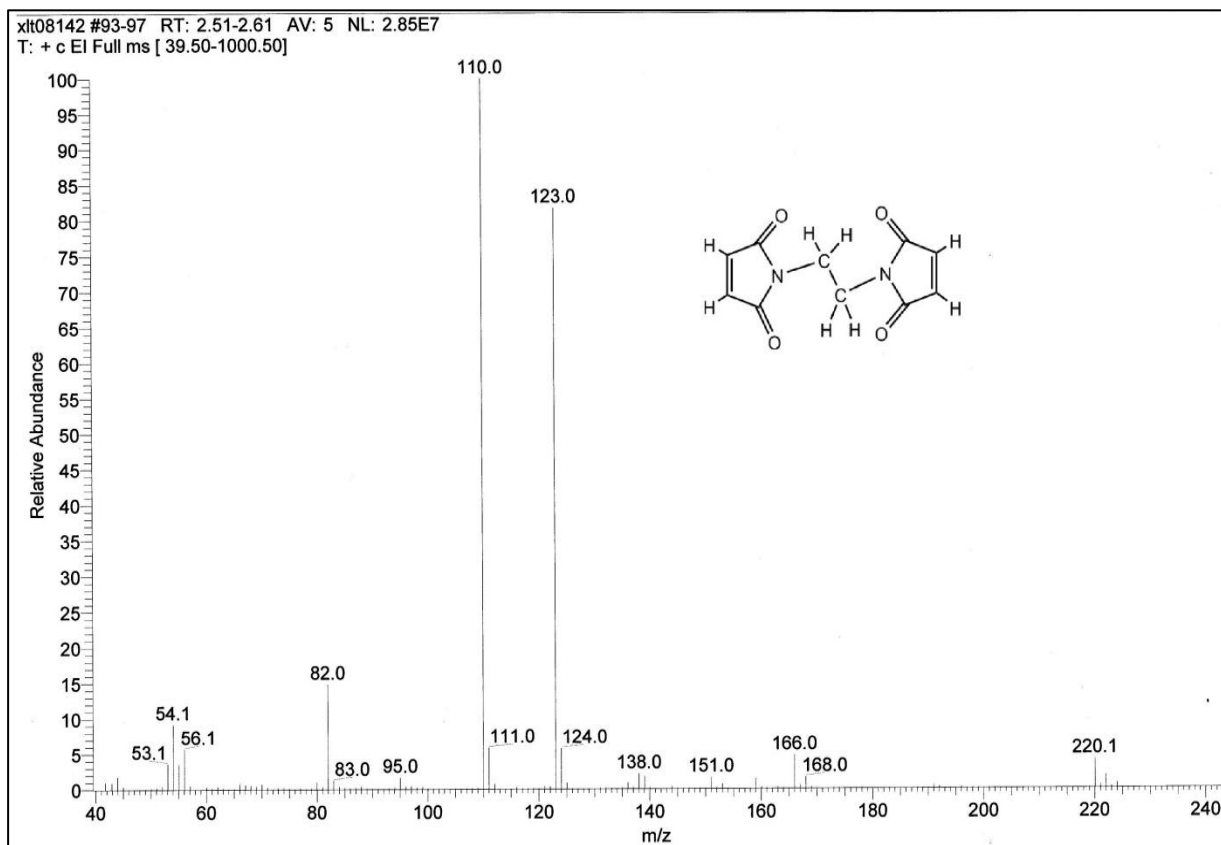


Fig.1 EI-Mass spectrum of compound BMI2.

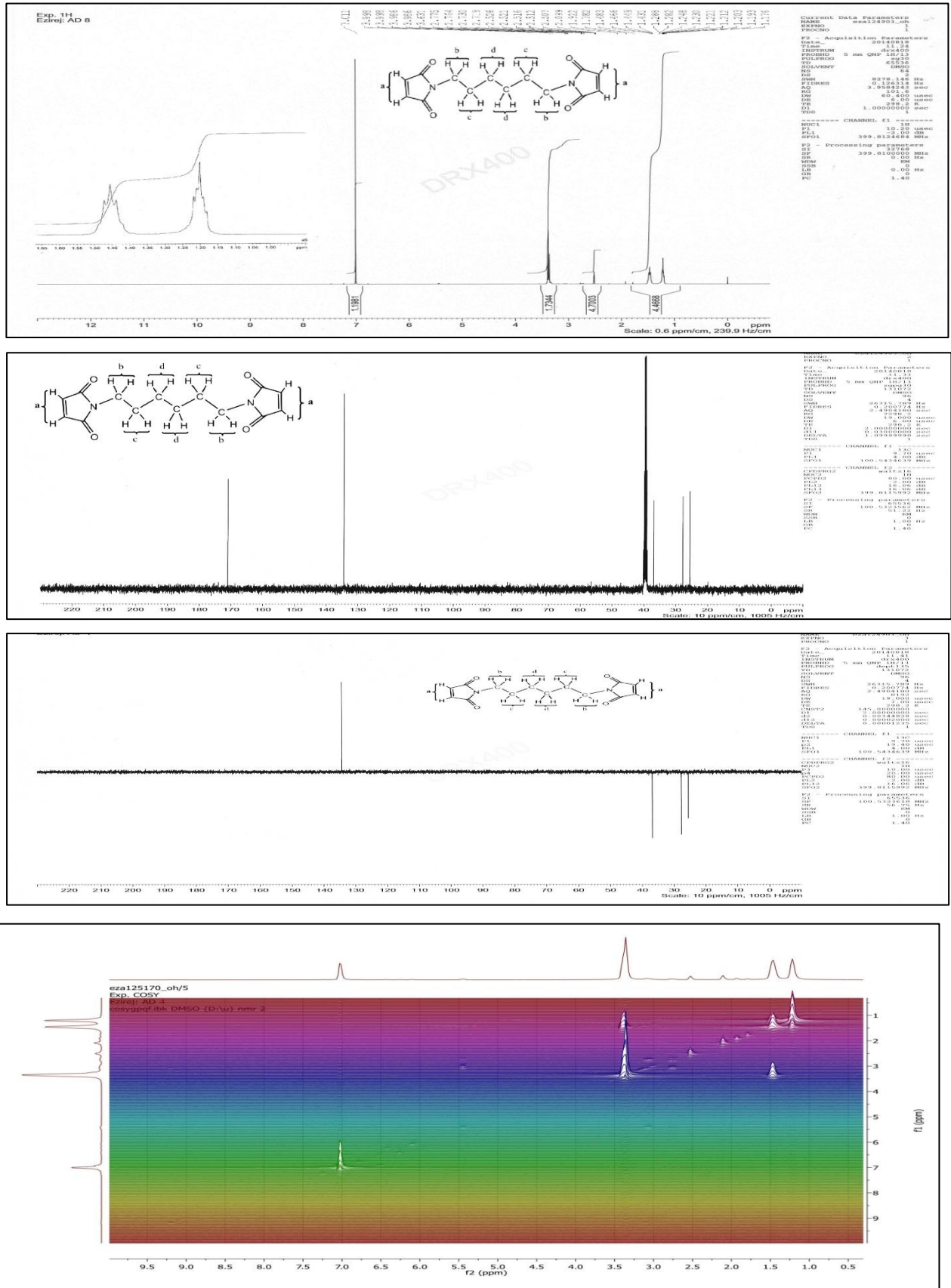


Fig.2 ¹HNMR, ¹³CNMR, Dept-135 and COSY spectra of compound BMI4

2-3 X-ray crystallography

A single crystal of the 1,1'-(sulfonylbis(4,1-phenylene)) bis (1H-pyrrole-2,5-dithione) (BMI7) product having colorless crystal crystallizes in the monoclinic system, space group $P2_1/c$ with the cell dimension $a = 11.698(3)$, $b = 15.002(3)$, $c = 20.053(6)$ Å and $\beta = 98.818(2)^\circ$ Volume = $3478.07(13)$ Å³, $R1 =$

0.0376 and $wR = 0.0866$. The crystal was measured on an Oxford Diffraction diffractometers (the firm is now called "Agilent"); either an "Xcalibur E" for Mo radiation or a "Nova A" (for Cu radiation), wavelength ($\lambda = 0.71073$ Å) radiation at 100(2) K.

2-4 Theoretical calculations.

The geometry optimization of prepared bismaeimides (BMI1-BMI7) were performed by carrying out density functional theory (DFT) calculations with Becke's three-parameter exchange and

Lee–Yang–Parr correlation functionals (B3LYP) with a combination of 6-311 G(d,p) basis set as implemented in Gaussian 09 suite of program [9]

3- Results and discussion

3-1 X-ray single crystal structure analysis

The structure of the compound 1,1'-(sulfonylbis(4,1-phenylene)) bis (1H-pyrrole-2,5-dithione) (BMI7) was also unambiguously confirmed by X-ray single crystal structure analysis. The colorless crystal crystallizes in the monoclinic system, space group $P2_1/c$ with the cell dimension $a = 11.698(3)$, $b = 15.002(3)$, $c = 20.053(6)$ Å and $\beta = 98.818(2)^\circ$ Volume = $3478.07(13)$ Å³, $R1 = 0.0376$ and $wR = 0.0866$.

The ORTEP diagram and the molecular structure optimization of compound (BMI7) is shown in Figures (1) and (2). The crystal data and structure refinement, hydrogen bonds and bond length and angles, were given in Tables 1-3.

The geometry calculated of compound (BMI1-BMI7) was optimized using a

Density functional theory DFT with B3LYP hybrid functional and 6-311G basis set, and is in good agreement with the structure obtained by the X-ray single crystal structure of compound BMI7 as in fig. 1.

The bond length of C(1)-C(2) is 1.393 Å, all aromatic bond length in range of 1.39 Å. C(8)-C(9) is 1.328 Å. Bond length for the C=C π bond exhibit acceptable values for C=C bond lengths [10]. The C-N bond length in range (1.400-1.419 Å) is significantly shorter than 1.4641 Å for a typical C-N single bond [11] which reflects an extent of π -conjugation with carbonyl group and aromatic ring. The C=O bonds, C(7)=O(3), C(10)=O(4) are 1.204 Å. The crystallographic values were very close to the theoretical values as in fig.3.

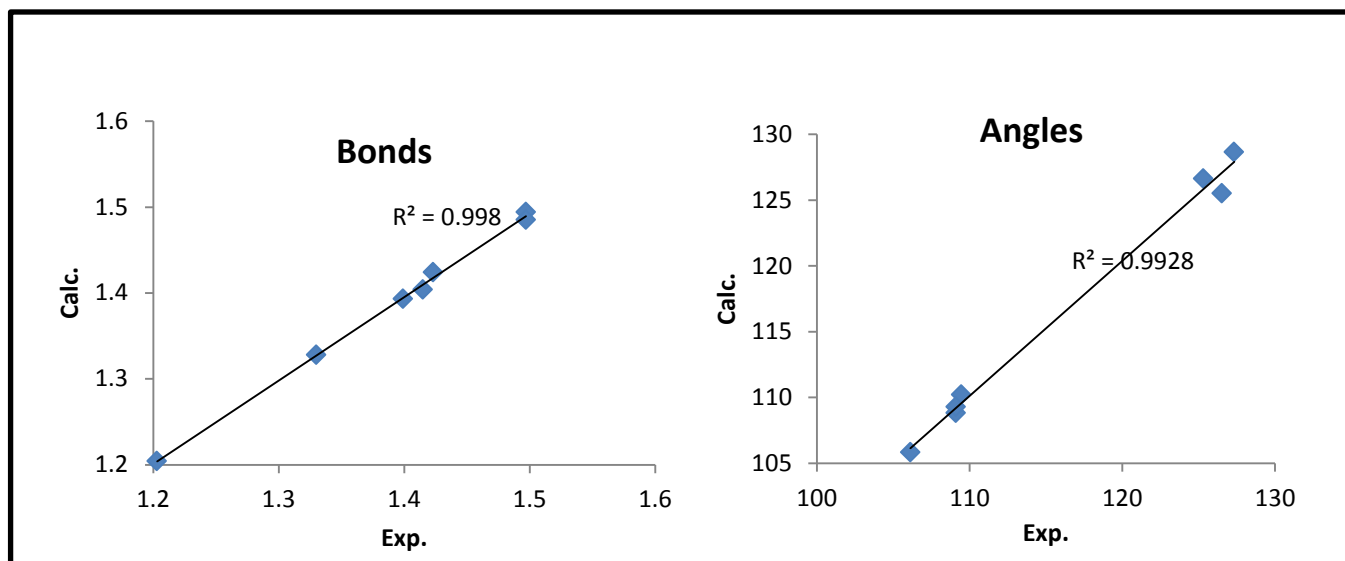


Fig.3 The bond lengths and bond angles obtained from crystal data of compound (BMI7) are comparable to theoretical data which calculated using DFT /B3LYP-6-311G (d,p) basis set.

Crystal structure study of many long-chain substances, is considerably hampered by the difficulty of growing single crystals large enough for a complete X-ray diffraction analysis.

The bond angles around the carbon atom C7 (i.e., O(3)-C(7)-N(1) = 125.53 ° (11), O(3)-C(7)-C(8) = 128.65°(12), N(1)-C(7)-C(8) = 105.82 ° (11), C(7)-C(8)-C(9) = 108.82 ° (11), C(8)-C(9)-C(10) = 109.29 ° (11). ORTEP plot of the compound 1,1'-(sulfonylbis(4,1-phenylene)) bis (1H-

pyrrole-2,5-dione) (BMI7) and Optimization of compound BMI7, calculated by DFT-B3LYP/6-311G(d,p) were given in figs. 4 and 5 respectively.

The bond lengths and bond angles obtained from crystal data of compound (BMI7) are comparable to theoretical data which calculated using DFT with B3LYP method and 6-311G (d,p) basis set. The selected bond lengths and angles of Compounds (BMI1-BMI7) are given in Table 4.

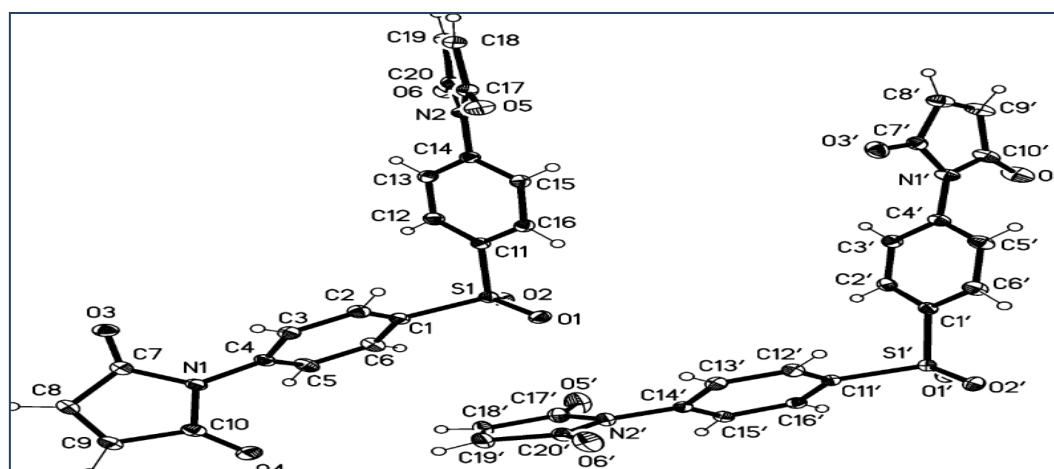


Fig. 4 ORTEP plot of the compound (BMI7).

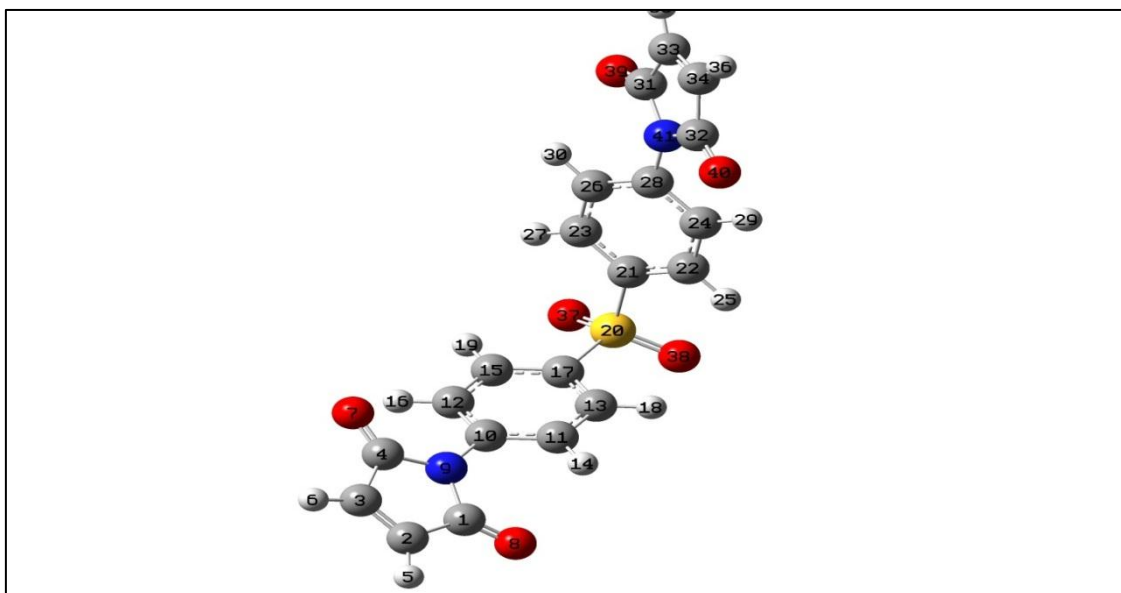


Fig.4 Optimization of compound BMI7, calculated by DFT-B3LYP/6-311G(d,p)

Table 1. Crystal data and structure refinement of compound BMI7

Identification code	adipla	
Empirical formula	C ₂₀ H ₁₂ N ₂ O ₆ S	
Formula weight	408.38	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁ /c	
Unit cell dimensions	a = 11.6988(3) Å	α = 90°
	b = 15.0024(3) Å	β = 98.818(2)°
	c = 20.0539(4) Å	γ = 90°
Volume	3478.07(13) Å ³	
Z	8	
Density (calculated)	1.560 Mg/m ³	
Absorption coefficient	0.231 mm ⁻¹	
F(000)	1680	
Crystal size	0.40 x 0.4 x 0.15 mm ³	
Theta range for data collection	2.22 to 30.85°	
Index range	-16<=h<=16, -21<=k<=21, -28<=l<=28	
Reflections collected	180867	
Independent reflections	10617 [R(int) = 0.0460]	
Completeness to theta = 30.50°	99.0%	
Absorption correlation	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.94932	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	10617 / 0 / 523	
Goodness-of fit on F ²	1.055	
Final R indices [I>2sigma(I)]	R1 = 0.0376, wR2 = 0.0866	
R indices (all data)	R1 = 0.0484, wR2 = 0.0916	
Largest diff. peak and hole	0.413 and -0.473 e.Å ⁻³	

Table 2. Hydrogen bonds [Å and °] from crystal data of compound (BMI7).

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(3)-H(3) ... O(1)#1	0.950	2.620	2.321(15)	131.300
C(5)-H(5) ... O(2)#2	0.950	2.620	3.158(15)	131.300
C(9)-H(9) ... O(3)#3	0.950	2.290	3.203(16)	116.300
C(12)-H(12) ... O(3)#1	0.950	2.390	3.112(15)	132.400
C(15)-H(15) ... O(4)#1	0.950	2.510	3.306(15)	141.600
C(19)-H(19) ... O(5)#1	0.950	2.640	3.277(17)	125.200
C(3')-H(3') ... O(1')#4	0.950	2.550	3.227(15)	128.700
C(8)-H(8) ... O(2')#5	0.950	2.300	3.178(16)	153.400

Symmetry transformation used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1, #2 x,-y+3/2,z+1/2, #3 -x,-y+1,-z+1, #4 -x,-y+2,-z+1, #5 -x,y-1/2,-z+3/2

Table3. The bond lengths [Å] and bond angles [°] obtained from crystal data of compound (BMI7) are comparable to theoretical data for compounds (BMI1-BMI7) which calculated using DFT /B3LYP-6-311G (d,p) basis set . The atoms are numbered depending on the numbering system in optimization geometry as in fig. 3

Geometric parameters	Compounds							
	BMI1	BMI2	BMI3	BMI4	BMI5	BMI6	BMI7	
							Calc.	Exp.
Bond (Å)								
C(4)-N(9)	1.444	1.395	1.396	1.396	1.397	1.412	1.415	1.404
C(10)-N(9)	-	1.454	1.460	1.459	1.459	1.426	1.423	1.424
C(4)=O(7)	1.194	1.206	1.208	1.207	1.207	1.204	1.203	1.204
C(4)-C(3)	1.491	1.507	1.503	1.504	1.504	1.498	1.497	1.494
C(3)=C(2)	1.322	1.332	1.331	1.332	1.332	1.330	1.330	1.328
C(2)-C(1)	1.491	1.502	1.501	1.504	1.504	1.498	1.497	1.485
Angle [°]								
C(4)-N(9)-C(10)	125.88	125.01	125.39	124.68	124.65	125.18	125.32	126.65
C(4)-N(9)-C(1)	108.23	110.64	110.42	110.64	110.63	109.63	109.46	110.22
O(7)-C(4)-N(9)	129.89	125.48	125.89	125.90	125.95	126.58	126.53	125.53
O(7)-C(4)-C(3)	124.17	128.58	128.07	128.11	128.07	127.28	127.33	128.65

3-2 FT-IR spectrum

The absence of the band at 3284 cm⁻¹ due to ν(N-H) amide and band at 3065 cm⁻¹ due to ν(O-H) absorption bands and appearance of band at 1694 cm⁻¹ for sym. ν(C=O) imide in the spectra of 1,1'-(ethane-1,2-diyl)bis(1H-pyrrole-2,5-dione) (BMI2) proving success of dehydration reaction [12]. The bismaleimides showed absorption band at range 3435cm⁻¹ attributed to amide groups [13].

Absorption bands of ν(C=C) and ν(C-N) imide appeared at 1585 cm⁻¹ and 1406 cm⁻¹ respectively. The asymmetrical and symmetrical stretching absorption band ν(CH₂) methylene groups, appeared at 2962, (2971-2898), (2940-2858), (2937-2848) cm⁻¹ in compounds BMI2, BMI3, BMI4, and BMI5 respectively and absence in compounds BMI1, BMI6, and BMI7 which haven't methylene group.

The position of the absorption bands vary depending on the type and length of the link spacer (R) between the two maleimide rings in the studied compounds (BMI1-BMI7). Absorption bands of $\nu(\text{C}=\text{O})$ has been shifted to a lower frequency compared , 1728-1689 cm^{-1} in compound from compound BMI1 (which direct bound with two maleimide rings) to compound BMI5 (which have octa methylene aliphatic chain spacer link between two maleimide rings). On the other hand, the C=O bond length of compound BMI1 is shorter than that of

another studied compounds, which are calculated using DFT. FT-IR spectra of all studied compounds shown in Fig.5. The absorption frequency of vinyl group increases with increasing methylene aliphatic chain spacer link between two maleimide rings, Figs. 6-8 show the correlation between some absorption bands and the type (direct link or aliphatic or aromatic) and length of aliphatic chain link spacer (R) substance between the two maleimide rings in the studied compounds (BMI1-BMI7).

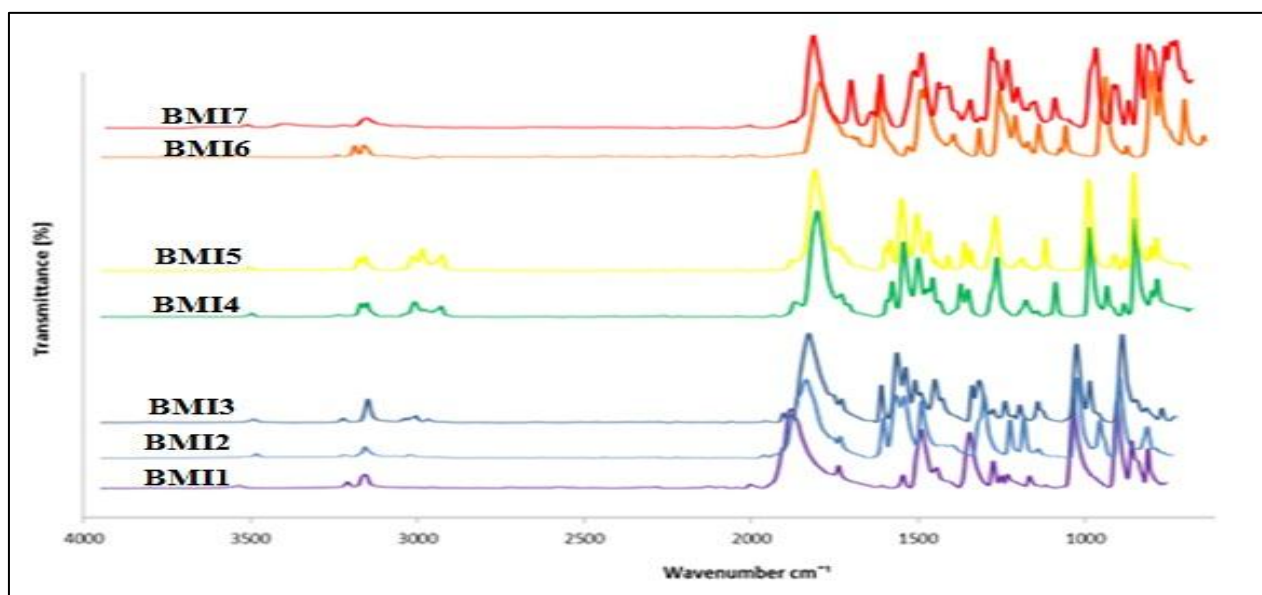


Fig.5. FT-IR spectra of Bismaleimides (BMI1-BMI7)

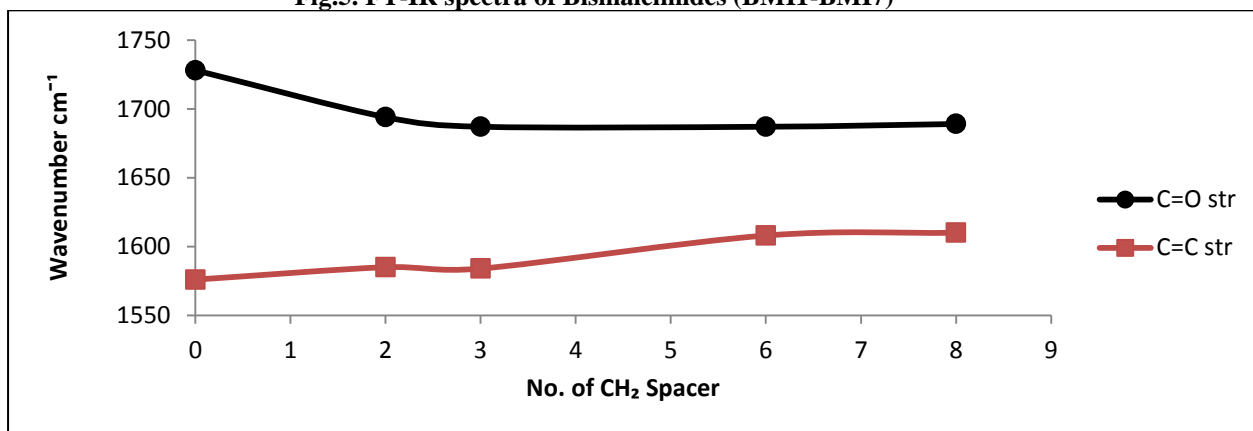


Fig. 6 The length of aliphatic spacer effect on the absorption bands of C=O and C=C in BMI1-BMI5

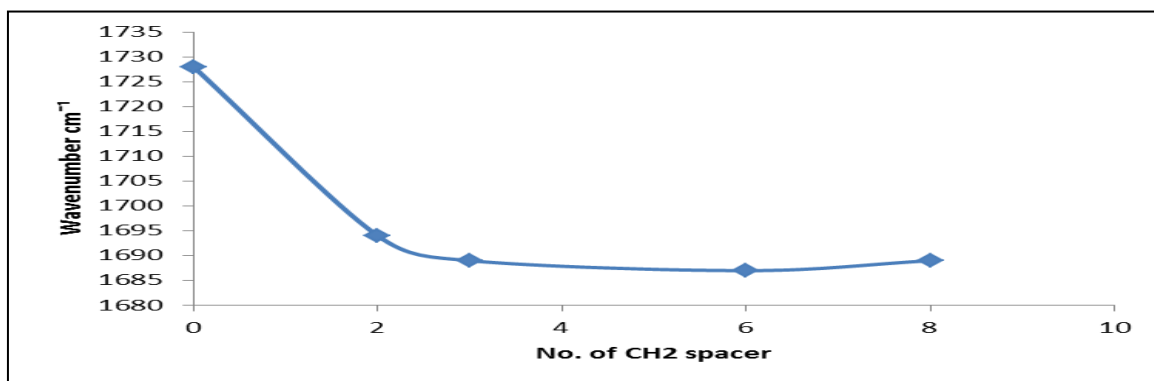


Fig. 7. The length of aliphatic spacer effect on the C=O absorption band of BMI1-BMI5

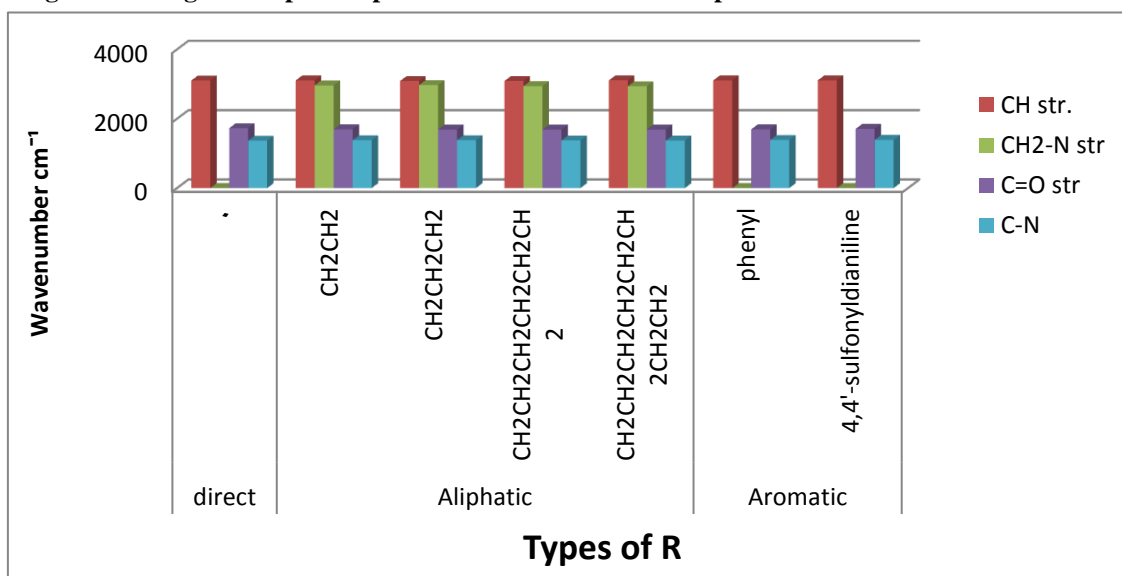


Fig. 5 The type and length of aliphatic spacer effects on the absorption bands of CH₂ str., CH₂-N str., C=O str. and C-N in BMI1-BMI7

4- Conclusions

A BMIs in different chain spacer linking between two maleimides rings were synthesis. The FT-IR observation showed that the position of the absorption bands varies depending on the type and length of the link spacer (R) between the two maleimide rings in the studied compounds (BMI1-BMI7). Absorption bands of $\nu(\text{C}=\text{O})$ have been shifted to a lower frequency compared , 1728-1689

cm^{-1} in compound from compound BMI1 (which direct bound with two maleimide rings) to compound BMI5 (which have octa methylene aliphatic chain spacer link between two maleimide rings). On the other hand, the C=O bond length of compound BMI1 is shorter than that of another studied compounds, which are calculated using DFT with B3LYP method and 6-311G(d,p) bases set.

Acknowledgements

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University of Braunschweig, Braunschweig, Germany.

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تحضير ودراسة الاطياف الاهتزازية والتركييب لبعض مركبات ثنائي المالمائيدات

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الخلاصة:

تضمنت هذه الدراسة تحضير ن،ن- الكيل (اريل) ثنائي المالمائيدات ودراستها طيفيا و باستخدام الانحراف بالاشعة السينية Single crystal X-ray لواحد منها . شخصت المركبات المحضرة باستخدام التآين الالكتروني في مطيافية الكتلة و طيف الاشعة تحت الحمراء و طيف الرنين المغناطيسي للبروتون والكاربون-13 و كذلك طيف الرنين المغناطيسي ثنائي الابعاد COSY و HSQC و HMBC ، كانت جميع المركبات مطابقة للتركييب المقترحة. اظهر طيف تحت الحمراء ان مواقع حزم الامتصاص تعتمد على نوع وطول السلسلة الرابطة بين حلقتي المالمائيد في المركبات المدروسة (BMI1-BMI7) . حزمة امتصاص مجموعة الكاربونيل ازيحت الى تردد اقل عند مقارنتها للمركبات من المركب BMI1 (الذي ترتبط فيه حلقتي المالمائيد بصورة مباشرة) الى المركب BMI5 (الذي ترتبط فيه حلقتي المالمائيد بسلسلة اليفاتية مكونة من ثمان مجاميع مثيل). من ناحية اخرى، طول الاصرة لمجموعة الكاربونيل للمركب BMI1 اقصر من المركبات المدروسة الاخرى والتي تم حسابها باستخدام نظرية دوال الكثافة DFT . تردد الامتصاص لمجموعة الفينائل تزداد بزيادة طول السلسلة الاليفاتية الرابطة بين حلقتي المالمائيد. تم دراسة التركييب الجزيئي للمركب BMI7 باستخدام الانحراف بالاشعة السينية X-ray وقورنت النتائج العملية مع النتائج المحسوبة نظريا باستخدام نظرية دوال الكثافة DFT وباسلوب B3LYP وعناصر قاعدة 6-311G(d,p) ، وقد استخدم لهذا العرض برنامج Gaussian 09 . اظهرت النتائج النظرية تقاربا مع تلك المقاسة عمليا.

الكلمات المفتاحية : ثنائي المالمائيدات ، ودراسة طيفية ، انحراف الاشعة السينية و نظرية دوال الكثافة.