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RESEARCH ARTICLE

Synthesis and Characterization of Substituted 1, 2, 4-triazole Pendant on Poly Ethylene via Microwave and Study Anticorrosion for its

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Abstract

New derivatives of 5-substited 1, 2, 4- triazole pendant on poly ethylene were obtained through this research. Aromatic acid hydrazide derivatives were prepared from reaction of different esters (prepared from different aromatic carboxylic acid with absolute ethanol in presence of H_2SO_4) with hydrazine hydrate in presence of ethanol as a solvent then reacted with poly acrylonitrile and poly acryl amide via microwave to give (4a-e) and (5a-e) compounds respectively. All prepared compounds were characterized by physical properties as (softening point, color and conversion \square), FT-IR, 1H -NMR for some of them and anticorrosion applications were measured.

Keywords: 1, 2, 4-triazole, Microwave, Anticorrosion.

Introduction

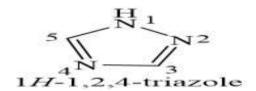
Heterocyclic is a very important branch of organic chemistry accounting for nearly one-third of modern publications. In fact two thirds of organic compounds are heterocyclic compounds. A cyclic organic compound having all carbon atoms in ring formation is referred to as carbocyclic compound. If at least one atom other than carbon forms a part of the ring system then it is designated as a heterocyclic compound.

Nitrogen, Oxygen and Sulfur are the common heteroatoms but heterocyclic rings containing other hetero atoms are also widely known [1]. In 1885, Bladin was the first scientist who gave the name triazole to the carbon nitrogen ring system (C₂N₃H₃) and described triazoles'

5 N^2 N_3 1H-1,2,3-triazole

1,2,4-Triazole derivatives have attracted scientists interest in having broad chemical properties, pharmalogical activities and synthetic versatility such as antifungal[7],

derivatives [2, 3]. There is significant and continuous concern in the chemistry of fivemembered N-heterocyclic compounds, mainly tetrazole (CH₂N₄), triazoles (C₂H₃N₃), and their substituted derivatives. Five-membered N-heterocyclic compounds are important structural fragments and considered as biologically active compounds [4]. Triazole is an important class of heterocyclic compounds exhibiting a wide range of pharmacological activities. Triazole, also known pyrrodiazole, is one of the classes of organic heterocyclic compounds containing a five membered diunsaturated ring structure composed of three nitrogen atoms and two carbon atoms at non-adjacent positions. Two isomers of triazole are [5, 6]:



antibacterial [8], anti-corrosive[9], antiinflammatory[10], anti cancer[11] and analgestic [12]. Moreover, there are many derivatives of 1, 2, 4-triazole used as the corrosion inhibitor in the corrosion inhibition of metals like copper, iron, aluminum, zinc. These compounds can be regarded as environment friendly inhibitors because of their characteristics of strong chemical activity and low toxicity. These derivatives are amphoteric in nature, forming salts with acids as well as bases, and have special affinity towards metal surface displacing water molecules on the surface. In addition, they possess abundant π -electrons and unshared electron pairs on the nitrogen atom that can interact with d-orbitals of any metal to provide a protective film. So research on 1,2,4- triazole derivative inhibitors has been a hot topic in recent years[13].

Material and Method

All used chemicals in this article were purchased from Sigma aldrich unless otherwise stated. Fourier transforms infrared radiation spectra were recorded SHIMADZU FTIR-8400 Fourier Transform Infrared spectrophotometer as KBr disc. ¹H nuclear magnetic resonance spectra were recorded on NMR spectrometer 400MHZ Avance 400 Bruker, Germany in central laboratory of Isfahan University/Tahran/Iran using tetra methyl silane as internal DMSO-d6 standard and solvents. as Softening point was determined on thermal microscope Riecherthermover.

Polymerization of acrylonitrile (PAN) [14]

In a polymerization bottle equimolar amounts (0.01 mol) of acrylonitrile was dissolved in (15ml) of freshly distilled dry THF. An amount equal to (0.05) % of the monomers weight of AIBN (initiator) was a dded then the bottle was purged with nitrogen and stoppered tightly. The clear solution was heated in a water bath maintained at (60°C) for 2hrs. The solution was poured into (25 ml) of methanol then the formed precipitate was filtered, washed with methanol several times and dried. The separated precipitate was purified by dissolved in DMF and reprecipitate from H₂O.

Polymerization of acryl amide (PAA)

In a polymerization bottle equimolar amounts (0.01 mol) of acryl amide was dissolved in (15ml) of freshly distilled dry THF. An amount equal to (0.05) % of the monomers weight of AIBN (initiator) was

added then the bottle was purged with nitrogen and stoppered tightly.

The clear solution was heated in a water bath maintained at (60) for 3 hrs. The solution was poured into (25 ml) of methanol then the formed precipitate was filtered, washed with methanol several times and dried. The separated precipitate was purified by dissolved in DMF and reprecipitate from H_2O .

Preparation of Ester Derivatives (2a-e) [15]

The aromatic carboxylic acid (5g, 0.03mol) was place in a round-bottom flask and dissolved in absolute ethanol (50 mL). Concentrated sulphuric acid (2 mL) and refluxed for 6 h. After cooling, carbon tetrachloride was added to the flask and extracted several times. The organic layer was washed with 5% sodium bicarbonate to remove unreacted acid. The solvent was then distilled off after drying with exsiccated sodium sulfate. The liquid so obtained was weighed and boiling point determined.

Preparation of Acid hydrazide Derivatives (3a-e)

A mixture of ester derivatives and hydrazine hydrate (0.05 mole) in 15 ml of ethanol was refluxed for 6 hrs. The content was concentrated, cooled and filtered. The separated precipitate was purified by dissolved in ethanol.

Preparation of 1,2,4-triazole Derivatives (4a-e)[16]

A mixture of poly acrylonitrile (3 mmol), hydrazide (1 mmol), and K_2CO_3 (0.5 mmol) in nBuOH (2mL) in a round-bottom flask was placed under microwave irradiation with normal absorption level and the temperature set to 150°C. After completion, the mixture was evaporated, diluted with MeOH and the separated precipitate was purified by dissolved in DMF and reprecipitate from H_2O .

Preparation of 1,2,4-triazole Derivatives (4a-e)[16]

A mixture of poly acryl amide (3 mmol), hydrazide (1 mmol), and K_2CO_3 (0.5 mmol) in nBuOH (2mL) in a round-bottom flask was placed under microwave irradiation with normal absorption level and the temperature set to 150°C. After completion, the mixture was evaporated, diluted with MeOH and the

separated precipitate was purified by dissolved in DMF and reprecipitate from H_2O .

Result and Discussion

In this research compound 5-substutited 1,2,4-triazole pendant on poly ethylene derivatives was prepared via microwave, the conversion ratio was (71-93)□ as shown in table (1), the measured softening point was [(165-185)-(215-235)] as shown in table (1), characterized by FT-IR spectrum as shown in table (2) and ¹H-NMR spectra of some prepared compounds as shown in (3). This research was carried out in several steps, the hydrazide derivatives [3a-e] synthesized from the reactions of ester derivatives [2a-e] with hydrazine hydrate in presence of ethanol as a solvent, which were carried out in Schemes (1).

FT-IR spectra of acid hydrazide derivative [3a] showed the appearance of some bands at [(3473 asy - 3392 sym), 3199, 1652 and 2950] cm⁻¹ belong to v (-NH2), v (-NH), v(C=O) amide and v(C-H) aliphatic respectively and disappearance band of v(C=O) ester. The compound [3a] reacted with poly acrylonitrile (PAN) and poly acryl amide (PAA) to form 5-substutited 1, 2, 4-triazole on poly ethylene [4a, 5a].

FTIR spectral of 1,2,4-triazole derivatives [4a,5a] showed disappearance of band of v(-NH2) and appearance of the bands at (1645) cm⁻¹ belong to v(C=N), and of [(3200), (1602), (3018), (2927)] cm⁻¹ belong to v(-NH triazole), v(C=C) aromatic, v(C-H) aromatic and v(C-H) aliphatic respectively for compound [4a]. And appearance of the bands at (1645) cm⁻¹ belong to v(C=N), and of [(3180), (1604), (3022), (2927)] cm⁻¹ belong to v (-NH triazole), v(C=C) aromatic, v(C-H) aromatic and v(C-H) aliphatic respectively for compound [5a] as shown in (Table 2). ¹H-NMR spectrum data of compound [4a] δ ppm in (DMSO) showed δ = 11.8(s, NH), 7.3-7.8(m, 4H) aromatic, 2.78(m, CH), 2.58(s, CH₃) 1.5(m, CH₂) as shown in (table 3).

FTIR spectrum of acid hydrazide derivative [3b] showed the appearance of some bands at [(3450 asy-3304 sym), 3223, 1660 and 2916] cm⁻¹ belong to v (-NH2), v (-NH), v(C=O) amide and v(C-H) aliphatic respectively and disappearance band of v(C=O) ester. The compound [3b] reacted with poly acrylonitrile (PAN) and poly acryl amide (PAA) to form 5-substutited 1, 2, 4-triazole on poly ethylene

[4b, 5b]. FTIR spectral of 1,2,4-triazole derivatives [4b,5b] showed disappearance of band of v(-NH2) and appearance of the bands at (1650) cm⁻¹ belong to v (C=N), and of [(3222), (1612), (3055-3097), (2871-2926)] cm-1 belong to v(-NH triazole), v(C=C) aromatic, v (C-H) aromatic and v (C-H) aliphatic respectively for compound [4b]. appearance of the bands at (1640) cm⁻¹ belong to v (C=N), and of [(3184-3210), (1608), (3033), (2860-2956)] cm-1 belong to v(-NH triazole), v (C=C) aromatic, v(C-H) aromatic v(C-H) aliphatic respectively compound for [5b] as shown in (table 2).

¹H-NMR spectrum data of compound [4b] δ ppm in (DMSO) showed δ = 11.8 (s, NH), 7.3-7.8(m, 4H) aromatic, 2.78(m, CH), 2.58(s, CH₃) 1.5 (m, CH₂) as shown in (table 3).FTIR spectrum of acid hydrazide derivative [3c] showed the appearance of some bands at [(3436 asy-3284 sym), 3186, 1645 and 2987] cm⁻¹ belong to v(-NH2), v(-NH), v(C=O) amide and v(C-H) aliphatic respectively and disappearance band of v(C=O) ester. The compound [3c] reacted with poly acrylonitrile (PAN) and poly acryl amide (PAA) to form 5-substutited 1, 2, 4-triazole on poly ethylene [4c, 5c].

FTIR spectral of 1,2,4-triazole derivatives [4c,5c] showed disappearance of band of v(-NH2) and appearance of the bands at (1654) cm^{-1} belong to v(C=N), and of [(3100-3182), (1595), (3004-3058), (2869-2935)] cm-1 belong to v(-NH triazole), v(C=C) aromatic, v(C-H) aromatic and v(C-H) aliphatic respectively for compound [4c]. And appearance of the bands at (1652) cm⁻¹ belong to v(C=N), and of [(3165), (1597), (3007-3049), (2850-2921)] cm-1 belong to v(-NH triazole), v(C=C) aromatic, v(C-H)aromatic and v(C-H) respectively for compound [5c] as shown in (table 2). ¹H-NMR spectrum data compound [4c] δ ppm in (DMSO) showed $\delta = 12.2(s, NH), 7.3-7.7(m, 4H)$ aromatic, 2.77(m, CH), 1.5(m, CH₂) as shown in table (3).

FTIR spectrum of acid hydrazide derivative [3d] showed the appearance of some bands at [(3431 asy-3398 sym), 3242, 1660 and 2947] cm⁻¹ belong to v (-NH2), v(-NH), v(C=O) amide and v(C-H) aliphatic respectively and disappearance band of v(C=O) ester. The compound [3d] reacted with poly acrylonitrile (PAN) and poly acryl amide (PAA) to form 5-substutited 1, 2, 4-triazole on poly ethylene [4d, 5d].

FTIR spectral of 1,2,4-triazole derivatives [4d,5d] showed disappearance of band of v(-NH2) and appearance of the bands at (1654) cm⁻¹ belong to v(C=N), and of [(3182-3200), (1596), (3028-3097), (2935-2970)] cm-1 belong to v(-NH triazole), v(C=C) aromatic, v(C-H) aromatic and v(C-H) aliphatic respectively for compound [4d].

And appearance of the bands at (1652) cm⁻¹ belong to v(C=N), and of [(3213), (1595), (3056), (2927)] cm-1 belong to v (-NH triazole), v(C=C) aromatic, v(C-H) aromatic and v(C-H) aliphatic respectively for compound [5d] as shown in (table 2). 1 H-NMR spectrum data of compound [4d, 5d] 8 ppm in (DMSO) showed δ =12.1(s, NH), 7.5-8.2(m, 5H) aromatic, 2.75(m, CH), 1.8(m, CH₂) for compound [4d] and δ =12.2(s, NH), 7.5-8.1(m, 5H) aromatic, 2.75(m, CH), 1.6(m, CH₂) for compound [5d] as shown in (table 3). FTIR spectrum of acid hydrazide derivative [3e] showed the appearance of some bands at [(3442 asy-3315 sym), 3222, 1660 and 2883]

cm⁻¹ belong to v (-NH2), v (- NH), v(C=O) amide and v(C-H) aliphatic respectively and disappearance band of v(C=O) ester.

The compound [3e] reacted with poly acrylonitrile (PAN) and poly acryl amide (PAA) to form 5-substutited 1, 2, 4-triazole on poly ethylene [4e,5e]. FTIR spectral of 1,2,4triazole derivative [4e, 5e]showed disappearance of band of v (-NH2) and appearance of the bands at (1641) cm⁻¹ belong to v (C=N), and of [(3218), (1600), (3062), (2935)] cm-1 belong to v (-NH triazole), v (C=C) aromatic, v (C-H) aromatic and v(C-H) aliphatic respectively for compound [4e]. And appearance of the bands at (1643) cm⁻¹ belong to v (C=N), and of [(3201), (1602), (3031-3060), (2927)] cm-1 belong to v (-NH triazole), v (C=C) aromatic, v(C-H) aromatic and v(C-H) aliphatic respectively for compound [5e] as shown in (table 2). ¹H-NMR spectrum data of compound [5a] δ ppm in (DMSO) showed δ =11.9(s, NH), 7.5-8.1(m, 5H) aromatic, 2.77(m, CH), 1.5 (m, CH₂) as shown in (table

$$\begin{array}{c} \textbf{n} \ \textbf{H}_2\textbf{C} = \textbf{CH} - \textbf{CN} \\ \hline \textbf{A1BN} \\ \hline \\ \textbf{(PAN)} \\ \hline \\ \textbf{n} \\ \textbf{(PAN)} \\ \hline \\ \textbf{n} \\ \textbf{(PAA)} \\ \hline \\ \textbf{(PAA)} \\ \hline \\ \textbf{(PAA)} \\ \textbf{(Sa-e)} \\ \textbf{(PAA)} \\ \hline \\ \textbf{(Sa-e)} \\ \textbf{(Sa-e)} \\ \textbf{(Sa-e)} \\ \textbf{(Sa-e)} \\ \hline \\ \textbf{(Sa-e)} \\ \textbf{(Sa-e)} \\ \textbf{(Sa-e)} \\ \hline \\ \textbf{(Sa-e)} \\ \textbf{(Sa-e)}$$

Scheme 1: preparation of compounds [(4a-e), (5a-e)]

Table 1: physical properties of the prepared compounds [PAN, PAA] [(4a-e),(5a-e)]

Comp	Compound	s. p. °C	Color	Yield %
No.	Structure	S. p. C	00101	11014 / 0
4a	HN N N N N N N N N N N N N N N N N N N	190-210	Orange	88
4b	HN N Me	210-230	Orange	93
4c	HN N CI	165-185	Orange	92
4d	HN N	205-225	Orange	90
4e	HN N	190-210	Orange	91
5a	HN N Me	185-205	Orange	71
5b	HN N Me	215-235	Orange	76
5c	HN N N CI	170-190	Orange	73
5d	HN N	200-220	Orange	75
5e	HN N	185-205	Orange	80

Table 2: FTIR spectral data of the prepared compounds [PAN, PAA] [2a-e] [3a-e] [(4a-e), (5a-e)]

Table 2: FIIR spectral data of the prepared compounds [PAN, PAA]					AA] [2a-e] [3a-e] [(4a-e), (5a-e)]					
Comp. No.	N=N	C=N	N-H	N-H2	C=O Ester	C=O Amide	C=C Aromatic	C-H aliphatic	C-H aromatic	C≡N
PAN								2937		2243
PAA				3387		1662	1618	2939	3195	
2a				•••	1719		1605	2979	3064	
2b				•••	1718		1612	2983	3170	
2c					1724		1607	2977	3087	
2d				•••	1721		1611	2968	3123	
2e				•••	1719		1603	2983	3064	
3a	•••	•••	3199	3473 asy 3392 sym		1652	1589	2950	3018	•••
3b	•••	•••	3223	3450 asy 3304 sym		1660	1616	2916	3031	•••
3c			3186	3436 asy 3284 sym		1645	1593	2987	3024	
3d			3242	3431 asy 3398 sym		1660	1618	2947	3014	
3e			3222	3442 asy 3315 sym		1660	1618	2883	3024	
4a(PAN)	1560	1645	3200	•••			1602	2927	3022	
4b(PAN)	1568	1650	3222			•••	1612	2871- 2926	3055-3097	
4c(PAN)	1564	1654	3100- 3182			•••	1595	2869- 2935	3004-3058	•••
4d(PAN)	1562	1654	3182- 3200			•••	1596	2935- 2970	3028-3097	
4e(PAN)	1552	1641	3218				1600	2935	3062	
4a(PAA)	1558	1645	3180	•••			1604	2927	3022	•••
4b(PAA)	1552	1640	3184- 3210			•••	1608	2860- 2956	3033	•••
4c(PAA)	1558	1652	3165				1597	2850- 2921	3007-3049	
4d(PAA)	1543	1652	3213	•••			1595	2927	3056	
4e(PAA)	1552	1643	3201	•••		•••	1602	2927	3031-3060	

Table 3: ¹H-NMR spectral data of some of the prepared compounds [4b, 4c, 4d, 4e, 5a, 5d]

Table 3: 'H-NMR spectral data of some of the prepared compounds [4b, 4c, 4d, 4e, 5a, 5d]							
Compound No.	Compound Structure	¹H-NMR spectral data δ ppm					
4b	HN N	11.8(s, NH), 7.3-7.8(m, 4H) aromatic, 2.78(m, CH), 2.58(s, CH ₃) 1.5(m,CH ₂)					
4c	HN N	12.2(s, NH), 7.3-7.7(m, 4H) aromatic, 2.77(m, CH), 1.5(m,CH ₂)					
4d	HN N	12.1(s, NH), 7.5-8.2(m, 5H) aromatic, 2.75(m, CH), 1.8(m,CH ₂)					
4e	HN N	11.9(s, NH), 7.5-8.1(m, 5H) aromatic, 2.77(m, CH), 1.5(m,CH ₂)					
5a	HN N N N N N N N N N N N N N N N N N N	11.8(s, NH), 7.3-7.9(m, 4H) aromatic, 2.70(m, CH), 2.50(s, CH ₃) 1.5(m, CH ₂)					
5d	HN N	12.2(s, NH), 7.5-8.1(m, 5H) aromatic, 2.75(m, CH), 1.6(m, CH ₂)					

Anticorrosion Application

The electrochemical corrosion data are shown in Table (4) like corrosion potentials (Ecorr), cathodics and anodicsd Tafel slopes (bc, ba) and corrosion current density (Icorr) get by of cathodic and anodic regions of the Tafel lines.

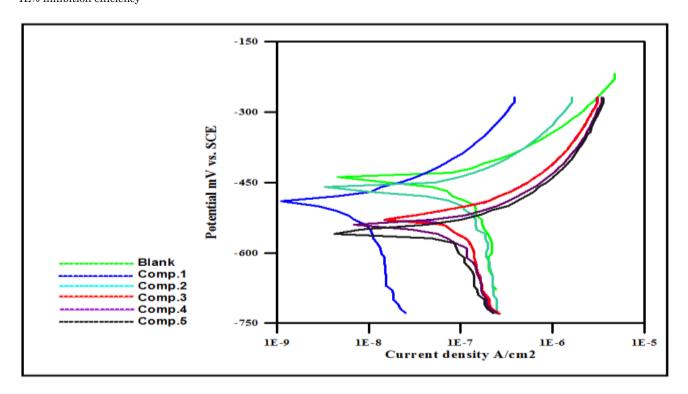
Figure 2 presents potentiodynamic polarization curves for C-steel in sea water containing). IE% was calculated in the equation below:

%IE = (Icorr (blank) – (icorr) / Icorr (blank)) × 100

Table 4: are showed good inhibition efficiency for all the compounds due to adsorption of the compounds with of Carbon steel in 3.5%NaCl whereas the compounds (4d,5d) were gave inhibition efficiency approximately same

Coating	-Ecorr (mV)	Icorr (μΑ/cm²)	-Bc (mV/Dec)	Ba (mV/Dec)	WL (g/m².d).d)	PL (mm/y)	IE%
Blank	-447.6	144.03	-747.5	123.2	36.0	1.67	-
4b Comp 3	-529.1	32.95	-91.7	59.9	8.24	0.382	77.12
4c Comp 2	-458.1	14.40	-60.6	36.9	3.60	0.167	90.00
4d Comp 1	-489.4	21.25	-73.7	41.6	5.31	0.247	85.24
5a Comp 5	-557.6	45.88	-121.2	71.8	11.5	0.533	68.14
5d Comp 4	-539.2	25.46	-88.3	38.0	6.37	0.296	83.32

Ecorr: corrosion potential Icorr: corrosion current density bc: cathodic Tafel slope ba: anodic Tafel slope WL: weight loss PL: pentration loss IE% inhibition efficiency



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