Rafidain Journal of Science

https://rsci.mosuljournals.com

Vol. 32, No.4, pp. 57-69, 2023

Synthesis and Characterization of some new Heterocyclic Polymer Compounds from Benzo[1,2-d:4,5-d] bis(thiazole)-2,6-diamine and [6,6'-Bibenzo[d]thiazole]-2,2'-diamine

Azhar H. Ali
Directorate of Education in Nineveh/ Ministry of Education/ Mosul / Iraq
Mohanad Y. Saleh **Khalid A. Owaid**
Department of Chemistry/ College of Education for Pure Science/ University of Mosul
Mohamed. A. Abdelzaher
Department of Environment Science and Industrial Development/ Faculty of Postgraduate Studies for Advanced Sciences/ Beni-Suef University/ Beni-Suef/ Egypt/ 62511

p-ISSN: 1608-9391
e -ISSN: 2664-2786

Article information

Received: 1/12/2022
Revised: 12/1/2023
Accepted: 26/1/2023

DOI: 10.33899/rjs.2023.181265

corresponding author:
Mohanad Y. Saleh
mohanadalallaf@uomosul.edu.iq
M. A. Abdelzaher
M.Abuelseoud@psas.bsu.edu.eg

ABSTRACT

In this study, A series of polyamides containing heterocyclic rings (A7-14) were synthesized from the reaction of two types of diamine monomers (benzo [1,2-d:4,5-d'] bis(thiazole)-2,6-diamine and [6,6'-bibenzo[d]thiazole]-2,2'-diamine) with four types of dicarboxylic acid chlorides. including the diamine monomers A5 and A6, which were prepared from the reaction of p-phenylenediamine or benzidine with Ammonium thiocyanate using bromine in glacial acetic acid, while the chlorides of dicarboxylic acids were prepared from the reaction of dicarboxylic acids with thionyl chloride. The resulted compounds were characterized by ATR-FTIR and HNMR spectrophotometry, and the thermal stability of the prepared polyamides was studied by TGA technique.

Keywords: Polymers, bis(thiazole), dicarboxylic acid, TGA .

INTRODUCTION

Chemical compounds that are produced on a large scale from natural or synthetic building blocks are known as industrial polymers. In this regard, the primary intermediate products used in an enormous range of applications made from a variety of materials, including common plastics, elastomers, thermosets, fibers, adhesives, and surface coatings (Arslan *et al*., 2019).

In 1929, Carothers divided synthetic polymers into two types based on the process used to create them: condensation polymers and addition polymers. In step-growth polymerization, also known as polycondensation, polymers are created by the reaction of two polyfunctional molecules and the removal of a minor molecule, such as water. In Fig. (1), typical condensation polymers are depicted. In a chain reaction, unsaturated monomers are converted to addition (or chain reaction) polymers (Goddard and Gruber, 1999). Addition polymer examples are shown in Fig. (1).

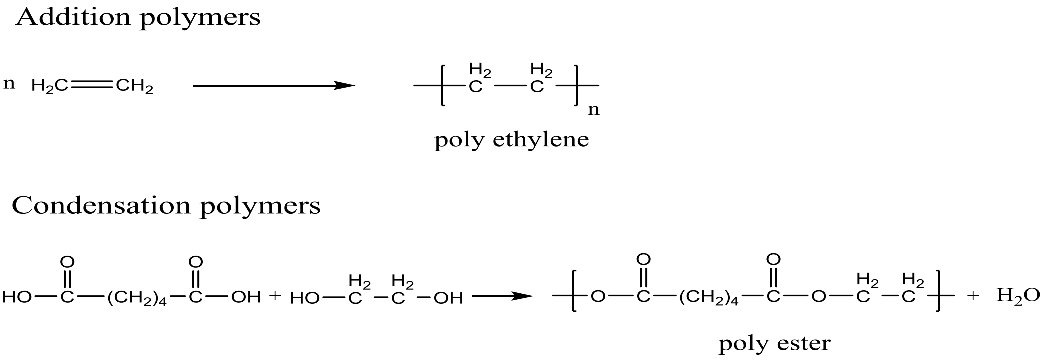
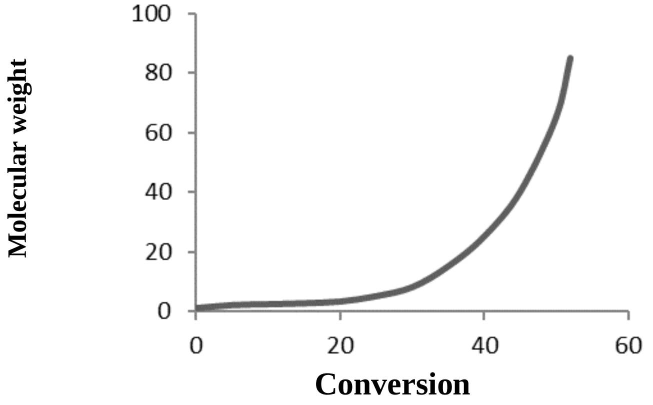


Fig. 1: Addition polymer

Step-growth polymerization begins with the interaction of two different monomers with opposite functional groups (each monomer possesses two similar and/ Or opposite functional groups at the same time for the functional groups of the second monomer) to form the dimer molecules that react with another dimer molecule to form a tetramer or with another monomer to form the trimer and so the process continues (Stille, 1981).

The natural gradual polymerization pathway is characterized by the fact that monomers, oligomers and polymers can interact with each other at any time since the interaction of functional groups is not highly dependent on the size of the molecules (Kricheldorf, 2014).

Therefore, the development of the average molecular weight is observed in the later stages of the reaction, as shown in the following figure:



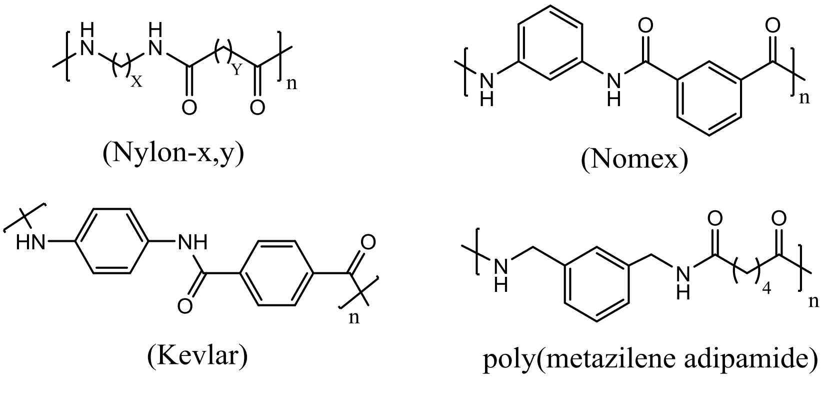
Polymers with recurrent amide groups (-CONH-) as essential components of the primary polymer chain are known as polyamides. The creation of polyamides by Carothers at Dupont in (1938), one of the earliest polycondensation polymers, led to their immense popularity as synthetic alternatives to natural fibers. Hexamethylenediamine and sebacoyl chloride are used to make them (Trossarelli, 2010). Different varieties of polyamides are now highly regarded for their strong mechanical strength and heat resistance.

Most polyamides are prepared by the conventional organic reaction (Amidation) of binary amines with binary organic acids, their esters, or their dichloride's, and this reaction is accompanied by the removal of (H_2O, ROH, or HCl) respectively as secondary products (Nakamura *et al.*, 2011).

Aliphatic or aromatic monomers, either acids or amines, can be used to produce polyamides. Aliphatic polyamides, which are typically hygroscopic can be made less so by adding an aromatic ring to their monomer structure. When polyamides are processed through injection molding, water can be a significant contaminant. Most frequently, polyamides are identified by the word Nylon followed by two digits with the first indicating the number of carbon atoms in the diamine monomer and the second the number of carbon atoms in the diacid monomer. If a cycle is present in either the amine or acid moiety of the polyamide, the first letter of the cycle name must also be included in the name. if an amino acid is used to make the polyamide, only one number is needed to describe the number of carbon atoms between the two functionalities (Saldivar-Guerra and Vivaldo-Lima, 2013).

Aliphatic polyamides (Nylon-x,y), have many industrial and textile applications due to their high mechanical strength, good durability and flexibility, so they are widely used in the production of clothing, ropes, rubber tires, carpets, pipes, wire insulation, membranes and adhesives.

Aromatic polyamides (aramids) such as a Nomex and Kevlar also have exceptional properties due to their hardness and high thermal mechanical properties, so they have been often used as substitutes for rubber and other commodities such as umbrellas, boats, thermoplastic tubes ... etc., and with that is, the aromatic polyamides have high melting points and poor solubility and are difficult to process and form, limiting their applications. In contrast, while aliphatic aromatic amides that contain di amine or di aromatic acid with a flexible aliphatic fraction such as poly (metazilene adipamide) Fig. (2), have the characteristics of Excellent and good solubility, in addition to having a higher melting point and glass transition temperature than nylon-6, nylon-6,6. and other mechanical properties (Ali and Kaneko, 2015), (Abdullah *et al.*, 2021), (Saeed *et al.*, 2023).



(Nylon-x,y)

(Nomex)

(Kevlar)

poly(metazilene adipamide)

Fig. 2: aliphatic and aromatic amides polymers

The newly fused heterocyclic compounds are of great interest in the field of medicinal chemistry because of their great contribution to the pharmacological and biological profile. Accordingly, 2-aminobenzothiazole Fig. (3) is one of the most important binaries fused heterocyclic organic compounds (Scheetz *et al.*, 1977), (Hamdoon *et al.*, 2022), (Saied *et al.*, 2022), has the molecular formula $C_7H_6N_2S$ and consists of a benzene ring fused with a five-membered thiazole ring (Jordan *et al.*, 2002), (Hussein *et al.*, 2022), (Daoud *et al.*, 2017), which contains a sulfur atom in position 1 and a nitrogen atom in position 3 (Venkatesh and Pandeya, 2009), (Sdeek *et al.*, 2021).

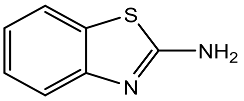


Fig. 3: 2-aminobenzothiazol

EXPERIMENTAL

All chemical compounds were provided by Fluka and BHD Company, and all melting points were evaluated using an electro thermal melting point device. Agilent Technologies 500 MHz was used to measure the 1HNMR and 13CNMR spectra of selected compounds in DMSO solvent, and Infrared spectrophotometer (ATR-FTIR) type Bruker was used for the IR measurements. The thermal stability of the prepared polymers was assessed using the analysis thermo gravimetric (TGA) technique in nitrogen atmosphere.

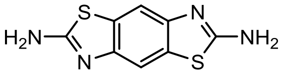
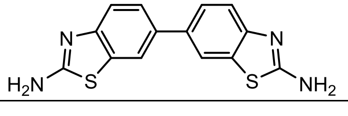
Synthesis of diacyl chloride derivatives (A_{1-4}) using dicarboxylic acids (Abbas *et al.*, 2020)

The dicarboxylic acid chloride monomers were prepared and used immediately and directly under dry conditions by refluxing the thionyl chloride with the dicarboxylic acids, where (0.05 mol) of the dicarboxylic acids were placed in the reaction flask, followed by the addition of (15 mL) of thionyl chloride slowly with cooling under conditions Dry, followed by refluxed of the reaction components at 70 °C until the complete dissolution of the dicarboxylic acids and making sure that no gases emitted (HCl and SO2), to obtain a white precipitate of terephthaloyl chloride, and products colorless liquid for each of glutaryl edipoyl and maleyl chloride.

Synthesis of benzo[1,2-d:4,5-d'] bis(thiazole)-2,6-diamine (A_5) and [6,6'-bibenzo[d]thiazole]-2,2'-diamine (A_6) (Hamed *et al.*, 2020), (Telvekar *et al.*, 2015).

Dissolve of (0.04 mol) of benzidine or p-phenylene diamine in (15 ml) of glacial acetic acid, then added, slowly add 0.29 mol of ammonium thiocyanate, dissolved in 70 ml of glacial acetic acid, to the mixture while stirring. then, Stir the mixture for 10 minutes.then cooled in an ice bath to (0 °C), after that add (6 ml) of bromine dissolved in (30 ml) of glacial acetic acid Dropped slowly through a separating funnel for half an hour, with stirring, Keep the temperature does not exceed 10°C. then the stirring continues for (8) hours at a temperature less than (25 °C), then the reaction mixture is left overnight in the refrigerator, after that (40 ml) of hot distilled water is added to the reaction mixture and heated to a temperature of (85-90 °C), then hot filtered, the precipitate is taken and 20 ml of acetic acid is added to it and heated to (90 °C) and hot filtered again. The filtrate is taken from two times and it is equalized with cooling with a concentrated ammonia solution. The formed precipitate is filtered, dried and washed with ethanol, knowing that the physical constants of the two compounds are shown in (Table 1).

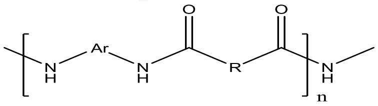
Table 1: The physical properties of the compounds ($A_{5,6}$)

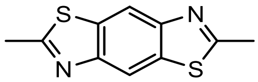
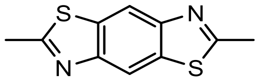
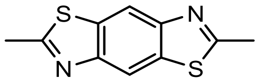
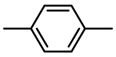
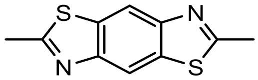
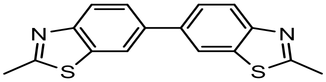
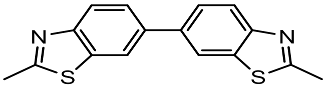
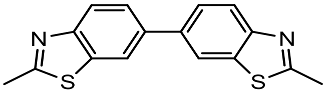
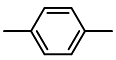
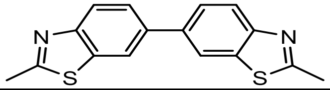
Comp No.	Ar	Molecular formula	m.p. (°C)	Yield (%)	Colour
A_5		$C_8H_6N_4S_2$	318-320	81	light green
A_6		$C_{14}H_{10}N_4S_2$	350>	90	Yellow

Synthesis of poly amide (A_{7-14}) (Telvekar *et al.*, 2015).

Dissolve (0.0035) moles of one of the compounds ($A_{5,6}$) in (10 ml) of dry (DMF) in a round two-hole flask of (100 ml) capacity, then add to it (1.5 ml) of pyridine. The solution is cooled in an ice bath. Then slowly added to it in the form of drops in an ice bath, (0.0035 mol) of one of the dicarboxylic acid chlorides (A_{1-4}) this dissolved in (5 ml) of dry dioxane. After the addition, the mixture was heated at a temperature (50 C) for one hour. With continuous stirring, then the reaction is left to cool at laboratory temperature, then (25 ml) of cooled water is added to it, the formed precipitate is filtered, washed with water well, and then left to dry, the physical constants of the polymers are shown in (Table 2).

Table 2: The physical properties of the compounds (A_{7-14})

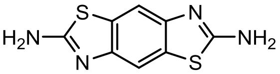
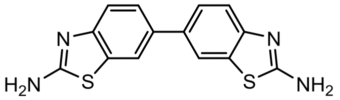


Poly. No	R	Ar	m.p. (°C)	Yield (%)	Colour
A_7	$(CH_2)_3$		>350(dec)	80	dark green
A_8	$(CH_2)_4$		215(dec)	55	greenish brown
A_9	HC=CH / \		>350(dec)	50	Black
A_{10}			>350(dec)	77	greenish yellow
A_{11}	$(CH_2)_3$		278(dec)	60	greenish yellow
A_{12}	$(CH_2)_4$		>350(dec)	66	Grey
A_{13}	HC=CH / \		>350(dec)	73	Brouwn
A_{14}			211(dec)	59	dark yellow

RESULT AND DESICCATION

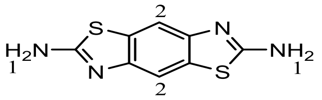
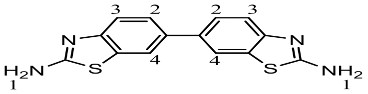
In this study, a number of polyamides were synthesized and characterized by the reaction of dicarboxylic acid chlorides (which were prepared and used immediately) with benzo[1,2-d:4,5-d'] bis(thiazole)-2,6-diamine. (A5) or [6,6'-bibenzo[d]thiazole]-2,2'-diamine (A6), which was prepared from the reaction of para-phenylenediamine or benzidine compounds with ammonium thiocyanate in glacial acetic acid in the presence of bromine. Characterization of the two compounds ($A_{5,6}$) by means of infrared and nuclear magnetic resonance of the proton, where the infrared spectrum of the two compounds ($A_{5,6}$) showed absorption bands at the range (1632-1633) cm^{-1} belonging to the group (C=N) within the thiazole ring, and the stretching bands. (C-S-C) within the range (941-1019) cm^{-1}, as well as the absorption bands of primary amine groups, which appeared within the range (3261-3348) cm^{-1}, and the spectrum showed aromatic stretching bands (C=C) at the frequency range (1414-1597) cm^{-1} and aromatic (C-H) bands at (3059-3091) cm^{-1}, as shown in (Table 3).

Table 3: The Spectral properties of the compounds ($A_{5,6}$)

Comp No.	Ar	IR spectrum, υ, cm^{-1}					
		NH_2	C=N	C-N	C-S-C	C-H Ar	C=C Ar
A_5		3269, 3348	1633	1414	941, 977	3059	1414-1589
A_6		3261, 3277	1632	1393	942-1019	3091	1443-1597

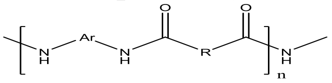
While the ^{1}HNMR spectrum gave single beams of primary amine protons (NH_2) in the range (7.24-7.36 ppm), and aromatic (C-H) protons in the range (7.45-7.95 ppm) as shown in the (Table 4).

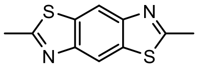
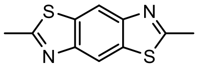
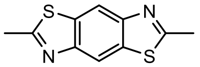
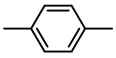
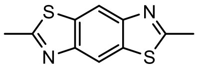
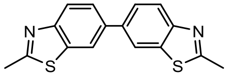
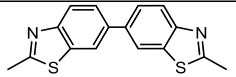
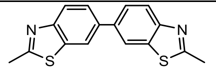
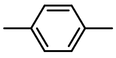
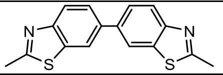
Table 4: The Spectral properties of the compounds ($A_{5,6}$)

Comp. No.	Structure	*^{1}H NMR (DMSO), ppm*
A_5		1: 7.24(s, 4H for NH_2), 2: 7.48(s, 2H for Ar-CH)
A_6		1: 7.36(s, 4H for NH_2), 2: 7.48(d, 2H for Ar-CH), 3: 7.50(d, 2H for Ar-CH), 4: 7.95 (d, 2H for Ar-CH)

Polyamides (A_{7-14}) were characterized by infrared spectroscopy (Saleh *et al.*, 2020), which showed the appearance of absorption bands at the range (1674-1693) cm^{-1} due to the stretching of amide carbonyl groups (NH-C=O), and absorption bands at the range (3150-3285) cm^{-1} belonging to (NH) amide groups. The spectrum showed aromatic (C=C) bands at the range (1444-1604) cm^{-1}, and aromatic (C-H) bands at the range (3049-3064) cm^{-1}, As well as the bands of stretching bonds (C=N) belonging to the thiazole ring, which appeared at the frequency range (1630-1659) cm^{-1}, as shown in (Table 5).

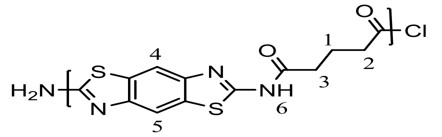
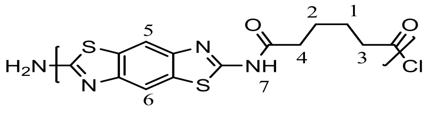
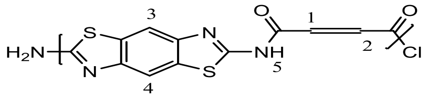
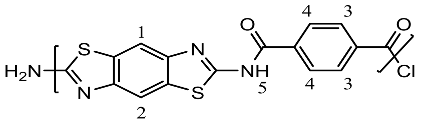
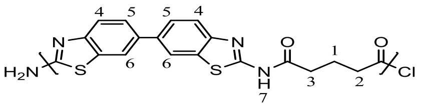
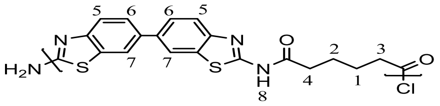
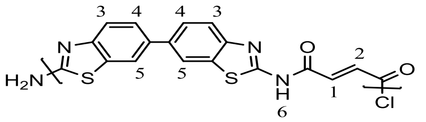
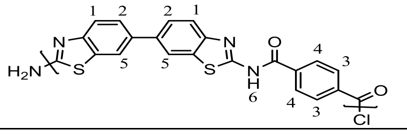
Table 5: The Spectral properties of the compounds (A_{7-14})



Poly No.	R	Ar	IR spectrum, υ, cm^{-1}					
			C=O amid	C=N	N-H amid	C-H Alp	C-H Ar	C=C Ar
A_7	$(CH_2)_3$		1693	1652	3181	2937	3058	1455-1591
A_8	$(CH_2)_4$		1690	1650	3191	2943	3067	1461-1592
A_9	HC=CH / \		1674	1644	3194	–	3056	1463-1591
A_{10}			1674	1630	3169	–	3060	1454-1593
A_{11}	$(CH_2)_3$		1685	1651	3150	2931	3049	1445-1603
A_{12}	$(CH_2)_4$		1686	1659	3181	2927	3056	1445-1602
A_{13}	HC=CH / \		1678	1633	3183	–	3061	1444-1602
A_{14}			1682	1632	3285	–	3064	1445-1604

The ^{1}HNMR spectrum of the polyamides (A_{7-14}) also showed the proton single bands of the amide (NH) groups in the range (12.37-13.3 ppm), and multiple peaks in the range (7.36-8.62 ppm) belong to the aromatic (Ar-CH) protons, as shown in the (Table 6).

Table 6: the Spectral properties of the compounds (A_{7-14})

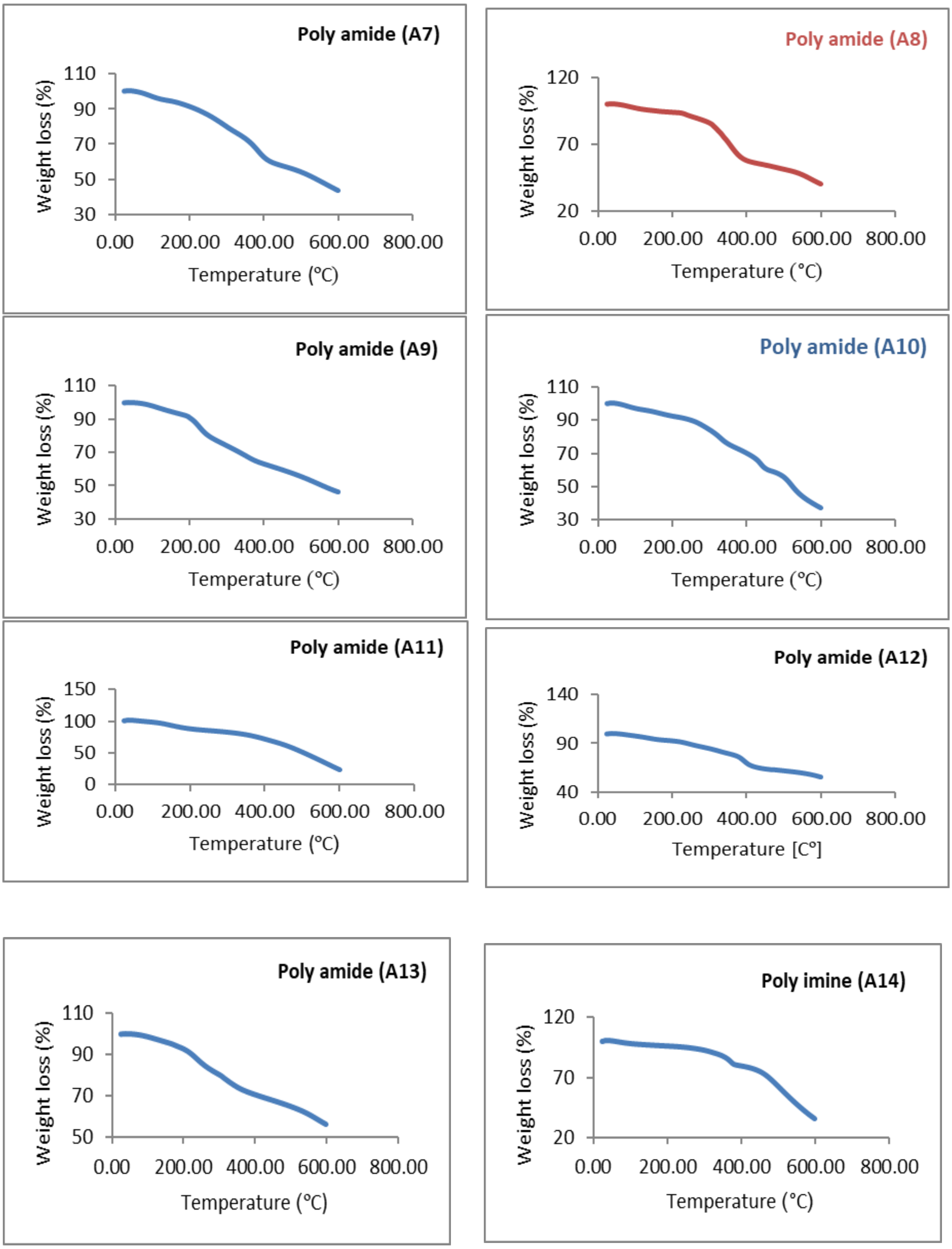
Poly No.	Structure	*^{1}H NMR (DMSO), ppm*
A_7		1: 1.84-1.90(p, 2H for CH_2), 2: 2.30-2.33 (t, 2H for CH_2), 3: 2.82-2.85(t, 2H for CH_2), 4,5: 7.77-8.52(m, 2H for Ar-CH), 6: 12.50 (s, 1H for NH-amide)
A_8		1: 1.48-1.58(p, 2H for CH_2), 2: 162-168(p, 2H for CH_2), 3: 2.25-2.28(t, 2H for CH_2), 4: 2.55-2.58(t, 2H for CH_2), 5,6: 7.77-7.84(d, 2H for Ar-CH), 7: 12.48(s, 1H for NH-amide)
A_9		1: 6.89(d, 1H for CH-en), 2: 7.25(d, 1H for CH-en), 3,4: 7.40-7.96 (m, 2H for Ar-CH), 5: 12.90(s, 1H for NH-amide)
A_{10}		1-4: 7.5-8.28 (m, 6H for Ar-CH), 5: 13.3(s, 1H for NH-amide)
A_{11}		1: 1.84-1.90(p, 2H for CH_2), 2,3: 2.30-2.33(t, 4H for CH_2), 5-7: 7.40-8.36(m, 6H for Ar-CH), 8: 12.37(s, 1H for NH-amide)
A_{12}		1: 1.51-1.59(p, 2H for CH_2), 2: 1.64-1.72(p, 2H for CH_2), 3: 2.25-2.28(t, 2H for CH_2), 4: 2.51-2.57(t, 2H for CH_2), 5-7: 7.4-8.37(m, 6H for Ar-CH), 8: 12.38(s, 1H for NH-amide)
A_{13}		1: 6.87(d, 1H for CH-en), 2: 7.23(d, 1H for CH-en), 3-5: 7.36-8.62 (m, 6H for Ar-CH), 6: 12.52 (s, 1H for NH-amide)
A_{14}		1-5: 7.37-8.60 (m, 10H for Ar-CH), 6: 13.27 (s, 1H for NH-amide)

Thermal properties

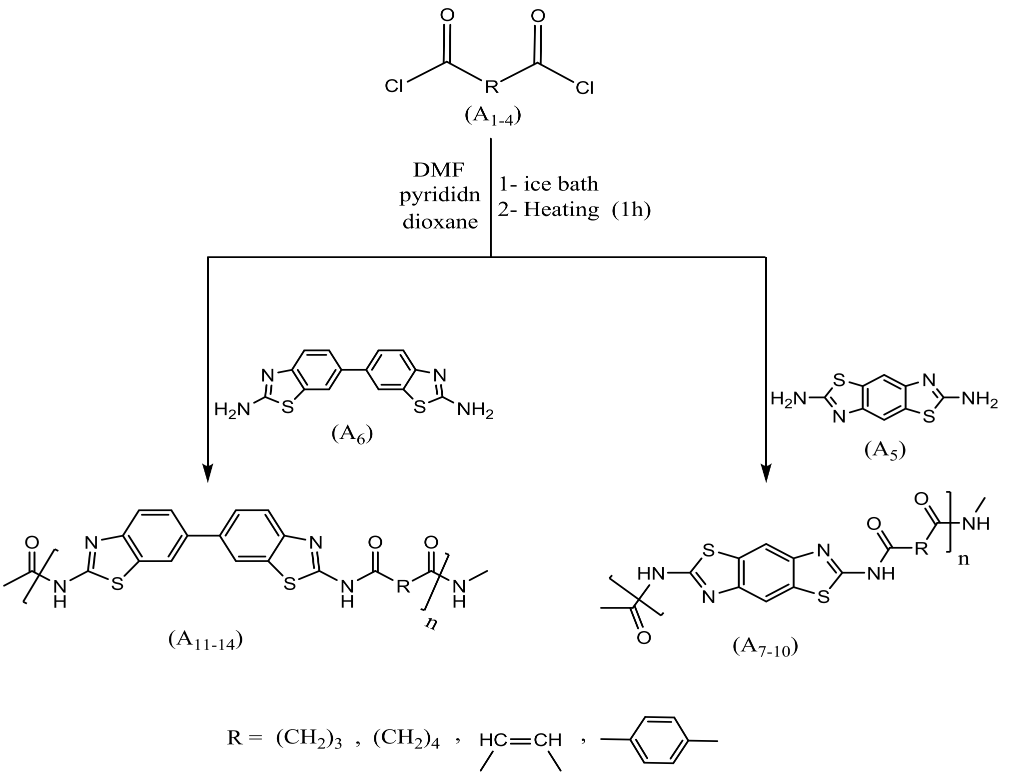
The thermal stability of the polymers (A_{7-14}) Was assessed by conducting a thermogravimetric analysis (TGA) under a nitrogen atmosphere at a heating rate of 10°C per minute. The polymers are heated to 600°C and monitoring the weight loss of the polymers (the decomposition ratio of the polymer) against the temperature.

The TGA curves showed that the polymers (A_{7-14}) have more than one slope point as shown in the thermogravimetric analysis curves below. The (TGA) curves indicate that the first weight loss for polymers (A_{7-14}) was at temperature 165, 197, 160, 210, 227, 192, 178 and 256°C versus weight loss by 7, 7, 6, 8, 14, 7, 6 and 6% respectively, which is due to the loss of water and solvents such as (DMF).

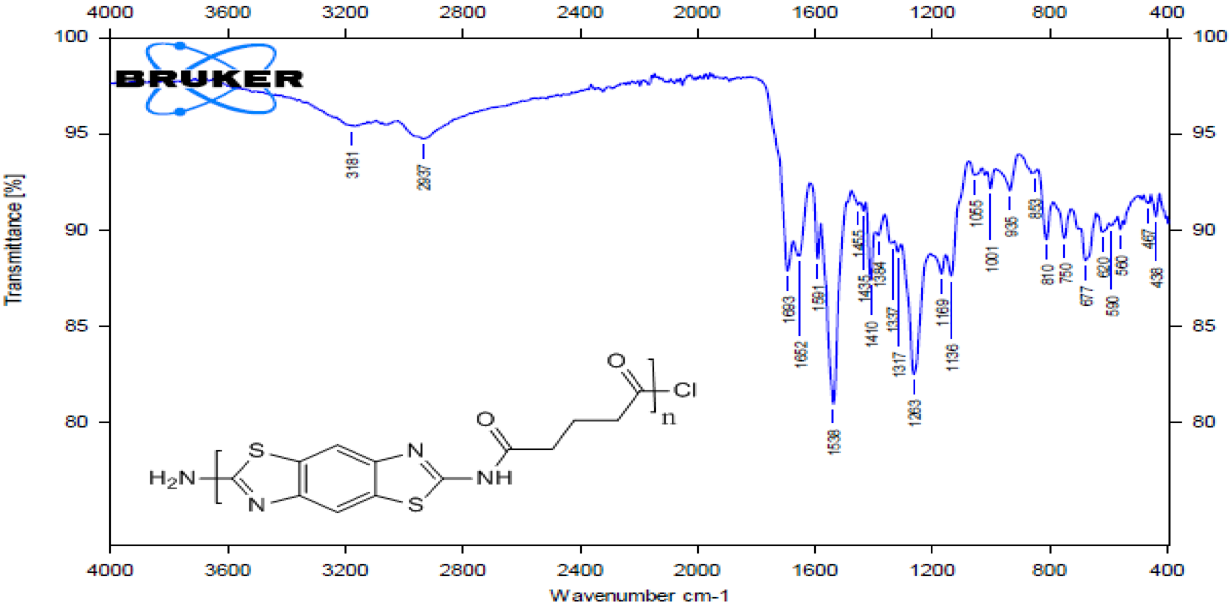
Whereas, the maximum thermal decomposition of polymers (A_{7-14}) was at 468, 511, 452, 463, 441, 528, 502 and 428 ° C, corresponding to a weight loss of 44, 49, 41, 41, 36, 41, 35and 23% respectively. The high thermal stability of polymers (A_{7-14}) is due to the high aromatic content of polymers that include heterogeneous aromatic rings between the polymeric chains, which are characterized by high thermal stability, in addition to the strong chemical bonding forces that link the polymeric chains and interlock the amide groups (Saied *et al*., 2021), (Kamil *et al*., 2015).



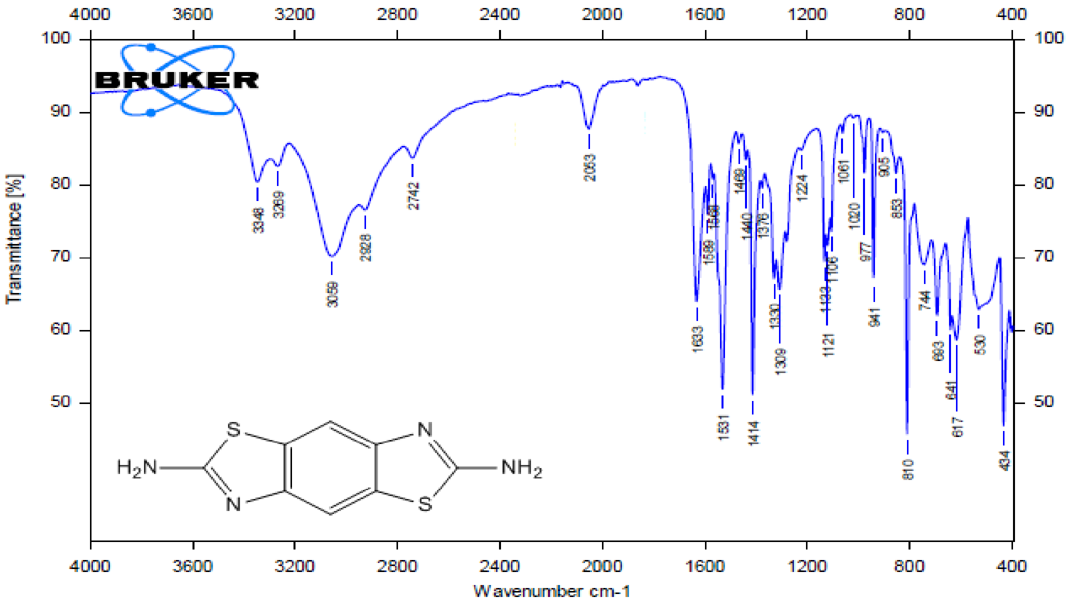
Thermogravimetric analysis curves of polyamides ($A_{7\text{-}14}$)



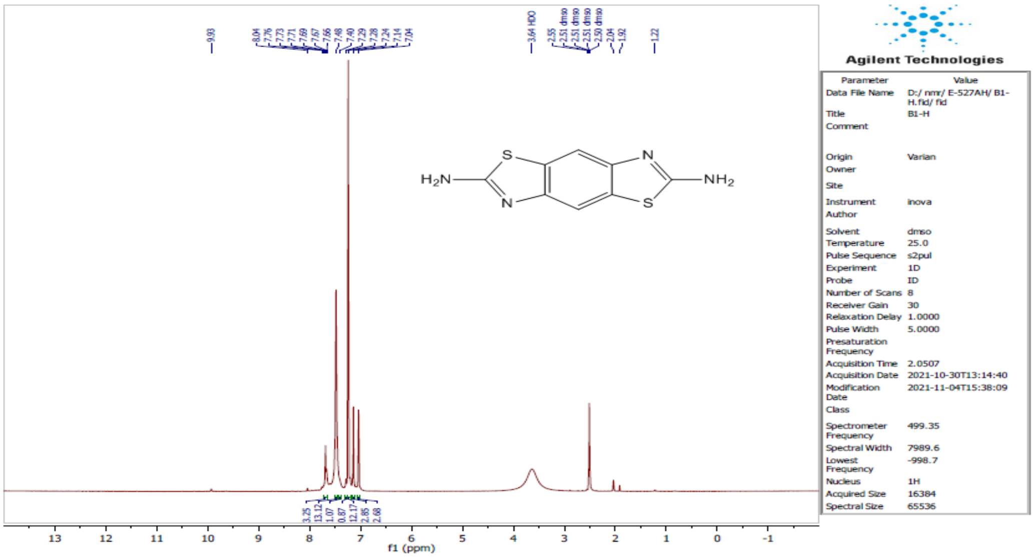
Scheme 1



IR spectrum of the compound (A_5)

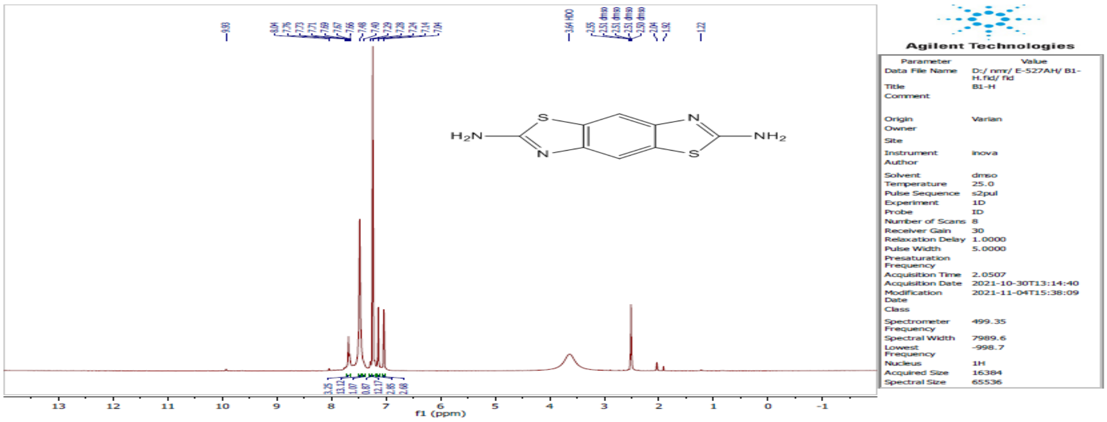


IR spectrum of the polyamide (A_7)



Parameter	Value
Data File Name	D:/ nmr/ E-527AH/ B1-H.fid/ fid
Title	B1-H
Comment	
Origin	Varian
Owner	
Site	
Instrument	inova
Author	
Solvent	dmso
Temperature	25.0
Pulse Sequence	s2pul
Experiment	1D
Probe	ID
Number of Scans	8
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	5.0000
Presaturation Frequency	
Acquisition Time	2.0507
Acquisition Date	2021-10-30T13:14:40
Modification Date	2021-11-04T15:38:09
Class	
Spectrometer Frequency	499.35
Spectral Width	7989.6
Lowest Frequency	-998.7
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

^{1}H-NMR spectrum of the compound (A_5)



Parameter	Value
Data File Name	D:/ nmr/ E-527AH/ B1-H.fid/ fid
Title	B1-H
Comment	
Origin	Varian
Owner	
Site	
Instrument	inova
Author	
Solvent	dmso
Temperature	25.0
Pulse Sequence	s2pul
Experiment	1D
Probe	ID
Number of Scans	8
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	5.0000
Presaturation Frequency	
Acquisition Time	2.0507
Acquisition Date	2021-10-30T13:14:40
Modification Date	2021-11-04T15:38:09
Class	
Spectrometer Frequency	499.35
Spectral Width	7989.6
Lowest Frequency	-998.7
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

^{1}H-NMR spectrum of the polyamide (A_9)

Electrical Conductivity

Polymers that possess conjugated double bonds are characterized by the non-centralization of their electrons and their movement along the chain compared to insulating organic molecules, because the polymer chain contains regions of high electronic density that are considered electron donors for the regions of low electronic density adjacent to them, and then the low ionization potential of electrons and the transmission of current at Length of polymeric chains and electrical conductivity (Khattab, 2007).

The volumetric electrical conductivity of polyamide (A_{10}) was measured, which gave an electrical conductivity that falls within the electrical conductivity of organic semiconductors, which amounted to (6.07E-10), where the volumetric conductivity was calculated by applying the following equation:

$\sigma = Id / AE$

whereas

σ = volumetric conductivity (S. cm-1)
I = current flowing in amperes (AMP)
d = disc thickness (cm)
E = potential difference measured in volts
A = area of the inner electrode used for measurement (influential area) (cm2)

CONCLUSIONS

Polymeric compounds are of great importance due to their ease of preparation. In this work, new polymeric compounds were synthesized from simple basic materials, and we obtained excellent results. Polymers with high heat content, stability and very good stability were obtained.

The obtained compounds were characterized using nuclear magnetic resonance spectra in addition to infrared spectroscopy. The synthetic polymers also gave excellent electrical conductivity compared to ionic semiconductors, even though they do not contain any metal within their basic composition.

REFERENCES

Goddard, E.D.; Gruber, J.V. (Eds.). (1999). “Principles of Polymer Science and Technology in Cosmetics and Personal Care”. CRC press. pp.1-2. https://doi.org/10.1201/9780203907948.

Abbas, A.M.; Mohammed Taib, A.S.; Saeed, N.H. (2020). Synthesis and characterization of linear thermally stable polyester contain Schiff bases. *Egyptian J. Chem.*, **63**(8), 2999-3013. https://doi.org/10.21608/ejchem.2020.25217.2526.

Abdullah, L.W.; Saied, S.M.; Saleh, M.Y. (2021). Deep eutectic solvents (Reline) and Gold Nanoparticles supported on Titanium Oxide (Au–TiO2) as new catalysts for synthesis some substituted phenyl (substituted-3-phenyloxiran) methanone enantioselective peroxidation. *Egyptian J. Chem.*, **64**(8), 4381-4389. https://doi.org/10.21608/ejchem.2021.68511.3498.

Ali, M.A.; Kaneko, T. (2015). “Polyamide Synthesis”. Encyclopedia of Polynanomeric Materials. Springer, Berlin, pp.1750-1762. https://doi.org/10.1007/978-3-642-29648-2_418.

Arslan, M.; Acik, G.; Tasdelen, M.A. (2019). The emerging applications of click chemistry reactions in the modification of industrial polymers. *Polymer Chem.*, **10**(28), 3806-3821. https://doi.org/10.1039/c9py00510b.

Daoud, K.M.; Saleh, M.Y.; Ismael, S.S. (2017). Synthesis and biological active of some substituted 1, 2, 4-Triazoles and their fused ring derivatives. *Diyala J. for Pure Sci.*, **13**(3-part 1). https://doi.org/10.24237/djps.1303.201c.

Hamdoon, A.M.; Al-Iraqi, M.A.; Saleh, M.Y. (2022). Synthesis of some multi-cyclic sulfhydryl donor compounds containing 1, 2-dithiol-3-thione moiety. *Egyptian J. Chem.*, **65**(3), 1-2. https://doi.org/10.21608/ejchem.2021.93344.4408.

Hamed, A.S.; Juber, A.K.; Khalil, A.M. (2020). Design, synthesis, characterization and biological evaluation of 6-Methoxy-2-aminobenzothioate derivatives against certain bacterial strains. *Egyptian J. Chem.*, **63**(10), 2-5. https://doi.org/10.21608/ejchem.2020.24849.2474.

Hussein, A.A.; Hamdoon, A.A.; Ahmed, S.S.; Mubarak, H.; Saleh, M.Y. (2022). Study of the effect of adding a blend of (ethylene vinyl acetate: styrene butadiene rubber) on the rheological properties of asphaltic materials. *Egyptian J. Chem.,***65**(11), 395-406. https://doi.org/10.21608/ejchem.2022.121325.5440.

Jordan, A.D.; Vaidya, A.H.; Rosenthal, D.I.; Dubinsky, B.; Kordik, C. P.; Sanfilippo, P. J.; Reitz, A. B. (2002). Potential anxiolytic agents. Part 4: novel orally-active N5-substituted pyrido [1, 2-a] benzimidazoles with high GABA-A receptor affinity. *Bioorg. Medic. Chem. Lett.*, **12**(17), 2381-2386. https://doi.org/10.1016/s0960-894x(02)00463-8.

Kamil, F.; Abid Hubeatir, K.; Shamel, M.; Al-Amiery, A.A. (2015). Microwave-assisted solvent-free synthesis of new polyimine. *Cogent Chem.*, **1**(1), 1075853. https://doi.org/10.1080/23312009.2015.1075853.

Khattab, A.F. (2007). Effect of polymeric chain flexibility on electrical conductivity of aromatic polyesters. *Raf. J. Sci.*, **18**(1), 20-27. https://doi.org/10.33899/rjs.2010.37317

Kricheldorf, H.R. (2014). "Polycondensation. History and New Results". Springer-Verlag Berlin Heidelberg, 1 p. https://doi.org/10.1007/978-3-642-39429-4.

Mohammed, E.R.; Saied, S.M.; Saleh, M.Y. (2022). Synthesis, characterization and biological evaluation study of Cephalexin (Ceph) and Isatin Schiff base and its complexation with some divalent metal ions. *Egyptian J. Chem.*, **65**(7), 5-4. https://doi.org/10.21608/ejchem.2021.106994.4914.

Nakamura, T.; Nagahata, R.; Takeuchi, K. (2011). Microwave-assisted polyester and polyamide synthesis. *Mini-Reviews in Organic Chem.*, **8**(3), 306-314. https://doi.org/10.2174/157019311796197454.

Saeed, Z.F.; Saleh, M.Y.; Sadeek, G.T. (2023). Synthesis and biological evolution of novel substituted 1, 2, 4-triazine from sulfanilic acid. *Egyptian J. Chem.*, **66**(1), 555-561. https://doi.org/10.2174/157019311796197454.

Saied, S.M.; Mohammed, S.J.; Khaleel, B.T.; Saleh, M.Y. (2021). Comparative studies between conventional techniques and green chemistry to synthesis of novel piperidinium salts ionic liquids (PBSILs). *J. Chem. Health Risks*, **11**(4), 451-456. https://doi.org/ 10.22034/JCHR.2021.686640.

Saied, S.M.; Saleh, M.Y.; Hamdoon, A.M. (2022). Multicomponent synthesis of Tetrahydrobenzo [a] xanthene and Tetrahydrobenzo [a] acridine derivatives using Sulfonated Multi-Walled Carbon Nanotubes as Heterogeneous Nanocatalysts. *Iranian J. Catalysis*, **12**(2), 189-205. https://doi.org/ 10.30495/IJC.2022.1955651.1924.

Saldivar-Guerra, E.; Vivaldo-Lima, E. (2013). "Handbook of Polymer Synthesis, Characterization, and Processing". Chap. 3. John Wiley and Sons. pp. 43-44, 48-49. https://doi.org/10.1002/9781118480793.

Saleh, M.; Ayoub, A.I.; Hammady, A.O. (2020). Synthesis biological studies of some new heterocyclic compound derived from 2-chloro-3-formyl quinoline and 4-(benzyl sulfonyl) acetophenone. *Egyptian J. Chem.*, **63**(12), 4769-4776. https://doi.org/10.21608/ejchem.2020.26354.2535.

Scheetz ME, I.I.; Carlson, D.G.; Schinitsky, M.R. (1977). Frentizole, a novel immunosuppressive, and azathioprine: their comparative effects on host resistance to Pseudomonas aeruginosa, Candida albicans, Herpes Simplex virus, and influenza (Ann Arbor) virus. *Infection and Immun.*, **15**(1), 145-148. https://doi.org/10.1128/iai.15.1.145-148.1977.

Sdeek, G.T.; Mauf, R.M.; Saleh, M.Y. (2021). Synthesis and identification of some new derivatives Oxazole, Thiazole and Imidazol from Acetyl Cysteine. *Egyptian J. Chem.*, **64**(12), 7565-7571. https://doi.org/10.21608/ejchem.2021.88755.4267.

Stille, J.K. (1981). Step-growth polymerization. *J. Chem. Education*, **58**(11), 862-866. https://doi.org/10.1021/ed058p862.

Telvekar, A.; Kelkar, S.; Mahanwar, P. (2015). Synthesis and characterization of new hetero ar-omatic polyamide from benzo[1,2-d:4,5-d'] bis(thiazole)-2,6-diamine. *International J. Scient. Engineer. Res.*, **6**(10), 303-307.

Trossarelli, L. (2010). The history of nylon. *Club Alpino Italiano, Centro Studi Materialie Tecniche, www. caimateriali. org/index.*

Venkatesh, P.; Pandeya, S.N. (2009). Synthesis, characterization and anti-inflammatory activity of some 2-amino benzothiazole derivatives. IJCRGG,**1**(4), 1354-1358. https://doi.org/10.1155/2009/645131.

تحضير وتشخيص بعض مركبات البوليمر الحلقية غير المتجانسة الجديدة من بنزو [1، 2–د :4، 5–د'] ثنائي (ثيازول) –2، 6–ديامين و [6،6'–بيبينزو [د] ثيازول] –2، 2'–ثنائي امين

أزهر حسين علي

مديرية تربية نينوى/ وزارة التربية

مهند يقضان صالح **خالد احمد عويد**

قسم الكيمياء/ كلية التربية للعلوم الصرفة/ جامعة الموصل

محمد ابو سعود عبد الظاهر

قسم علوم البيئة والتطوير الصناعي/ كلية الدراسات العليا للعلوم المتقدمة/ بني سويف/ مصر

الملخص

في هذه الدراسة، تم تحضير عدد من البولي أميدات (A7-14) التي تحتوي على حلقات حلقية غير متجانسة من تفاعل نوعين من مونومرات ثنائي الأمين (بنزو [1، 2–د: 4، 5–د '] ثنائي(ثيازول) –2، 6–ديامين و [6،6'–بينزو [د] ثيازول] –2،2'–ثنائي امين) مع أربعة أنواع من كلوريد حمض الكربوكسيل، حيث تم تحضير مونومرات ثنائي أمين(A5 ، 6) من تفاعل 1، 4– بنزين ثنائي الأمين أو بنزيدين مع ثيوسيانات الأمونيوم. في حامض الخليك الثلجي بوجود البروم، بينما تم تحضير كلوريدات أحماض ثنائي الكربوكسيل من تفاعل أحماض ثنائي الكربوكسيل مع كلوريد الثيونيل. تم تشخيص المركبات المحضرة باستخدام طيف الأشعة تحت الحمراء(ATR-FTIR) ومطياف الرنين المغناطيسي النووي (^{1}HNMR)، وتمت دراسة الثبات الحراري للبولي أميدات المحضرة بتقنية التحليل الحراري الوزني (TGA).

الكلمات الدالة: البوليمرات، ثنائي (ثيازول)، حامض ثنائي الكربوكسيل، المحتوى الحراري للبوليمرات.