ISSN: 0975 -8542

Journal of Global Pharma Technology

Available Online at: www.jgpt.co.in

RESEARCH ARTICLE

Synthesis and Characterization of Heterocyclic Compounds and Polymers with Studying the Biological activity for some of them

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Abstract

This paper deals with the preparation of new monomers and polymers which including heterocyclic unit. The diacid chlorides compounds [1-3] were prepared from the reaction of glutaric acid, adipic acid, terephthalic acid with thionyl chloride. Succinic acid reacted with ethanol to produce compound [4]. Compound [4] reacted with hydrazine hydrate to obtain succinic hydrazide [5].Compound [5] reaction with CS_2 and KOH in absolute ethanol to produce compound [6].The polymers [7-12] have been created by reacting diacid chlorides compounds [1-3] with compound[5] or [6] in dry pyridine with some drops of DMF. The topology of produced compounds has characterized through their spectral and analytical data as in FT-IR spectra, Thermal analysis [DSC, TGA] and Antibacterial activity investigated using dual bacteria kinds.

Keywords: *Succinic hydrazides, Oxadiazole, Heterocyclic polymers.*

Introduction

Heterocyclic chemistry stands for highly imperative division of organic chemistry with about one-third of recent studies in the literature especially for heterocyclic compounds. A cyclic organic compound with each carbon atom in ring creation has known as carbocyclic compound. In the case of as a minimum single atom different than carbon arrangements, a portion of the ring system at that point is termed as a heterocyclic compound[1]Oxygen, Sulfur with Nitrogen stand for the highly conventional heteroatoms. Nevertheless, heterocyclic rings having different heteroatoms are likewise extensively identified. A massive amount of heterocyclic compounds have recognized in an increasing number and swiftly [2] Oxadiazoles represent 5 rings compounds by means of 3 hetero atoms (single O atom along with dual N atoms).The oxadiazole ring possesses 4 isomers[3]as presented in Figure (1).

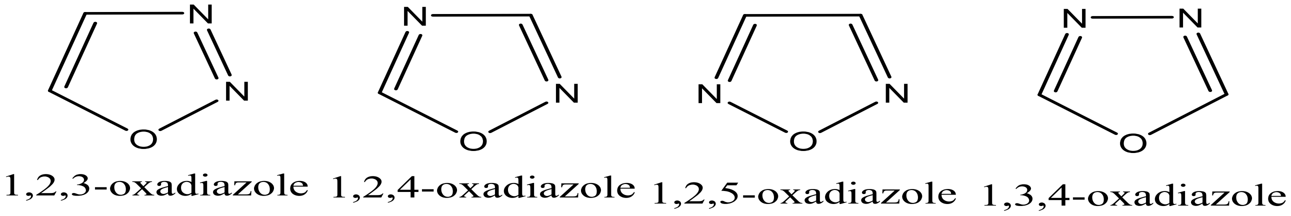


Figure 1: oxadiazole ring isomers

1, 3, 4-Oxadiazole stand for highly thermally stable isomer that has distinct consideration .This is principally as a result of the enormous applications in numerous areas. The steadiness of 1, 3, 4-oxadiazole boosted through alkyl along with aryl substituting on 2 and 5 positions. The substituted aryl of 1, 3, 4-oxadiazole have a smaller amount of sensitivity to acid as compared with alkyl-substituted. The hydrolysis susceptibility increases as solubility increases based on the sequence illustrated in Figure (2) [4]:

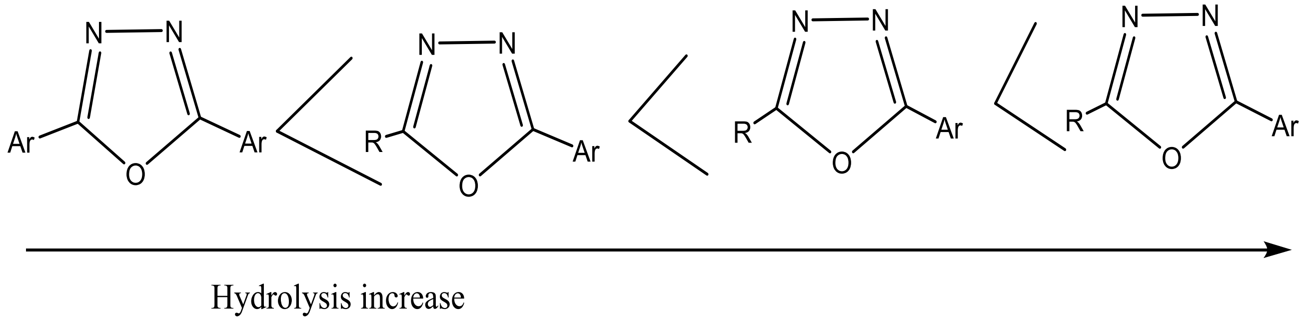


Figure 2: Substituted 1, 3, 4-oxadiazole trend headed for hydrolysis

The 1, 3, 4-Oxadiazole are organically multipurpose compounds involving fungicidal, bactericidal, analegisc, herbicidal, anti-inflammatory, hypoglycemic and transqulizing agents. Additionally, countless 1,3,4-oxadiazoles have been appropriate for usages in scintillation materials, photography, corrosion inhibitors, dye industry along with thermal stabilizers for riged polyvinyl chloride [4, 6].They represent high linear polymers including heterocyclic rings or rings group associated with through solitary or more covalent bonds. Like these polymers as a group are frequently instinctively rigid and characteristically resilient to thermal degradation.

Experimental

Materials

All employed chemicals in the synthesis process were delivered by BDH

Instrumentation

Melting points have been read by means of hot stage Gallen Kamp apparatus and they have been uncorrected. IR spectrums have been investigated by KBr disc based on SHIMADZU-FT-IR-8400 spectrometer.

Synthesis Compounds

Preparation of Adipoyl, Glutaryl and Terephthayl Chloride [1-3]

Compounds [1-3] were prepared as described as in [7]

Synthesis of Diethyl Succinate [4][8]

A succinic acid (0.001 mol) has mixed with 20 ml absolute ethanol. After that, about 10ml of concentrated sulfuric acid was inserted. This mixture has been refluxed for 4hours. Subsequently, the mixture was poured in 50 ml of distilled water and extracted with ethyl acetate, with water cleaned and at that time the organic layer was dehydrated. Accordingly, the solvent has evaporated.

Synthesis of Succinohydrazide [5][9]

A mixture of compound [4] (0.01 mole) with (10 ml.) of Hydrazine hydrate 80%. The projected mixture has refluxed for 6 hours. The product has been cooler as compared with room temperature and filtered, washed with ethanol. The final product was dried under m.p 125℃.

Synthesis of Compound [10][6]

A compound mixture based on [5] with (0.015 mol) and 10ml absolute ethanol, then (1.5 ml) of carbon disulfide CS_2 with (0. 6 gm) KOH was added. This mixture was refluxed in water bath for 10h. The resulted crude has dissolved in distilled water and acidified with hydrochloric acid. The solid product has finally filtered.

Synthesis of Polymers [7-12]

Compound [5] or [6] (0.0068 mol) was dissolved in dry pyridine (5ml) with some drops of DMF. The mixture was cooled and compounds [1-3] (0.0068mol.) Was added drop wise with stirring to this mixture. The stirring was continued for 24 hrs. Then, the mixture has transferred to ice water with (5ml) of concentrated hydrochloric acid. The precipitous item has filtered and dried.

Results and Discussion

Compound [5] was prepared from the reaction between diethyl succinate and hydrazine hydrate in ethanol. The organizational consignment of the product has been depended on its melting point and spectral data. The FT-IR band of compound [5] as in figure (3) has characterized by the disappearance of carbonyl of ester and appearance of $NHNH_2$ group at (3313, 3201)cm^{-1} besides the presence of carbonyl of amide at 1645 cm^{-1} [11]. Compound [6] was synthesized from reaction compound [5] with CS_2 and KOH in absolute ethanol. FT-IR result of compound [6] showed disappearance of $NHNH_2$ group and carbonyl of amide at (3313, 3201) cm^{-1} as well as (1645) cm^{-1} respectively with appearance of (C=N), (C-O) at (1620 ,1639) and (1138)cm^{-1} respectively [12]. Polymers [7-12] were synthesized by reacting compound [5] or [6] with compounds [1-3] in dried pyridine with drops of DMF. The structures of these polymers have detected by FT-IR spectrum. FT-IR results of compound [9] , illustrated the presence of aromatic (C-H) at (3062) cm^{-1}, alph (C-H) by (2872,2943)cm^{-1}, (C=C) band by (1571) cm^{-1} and carbonyl groups by (1674)cm^{-1} with disappearance of (C=O-Cl) group [13].

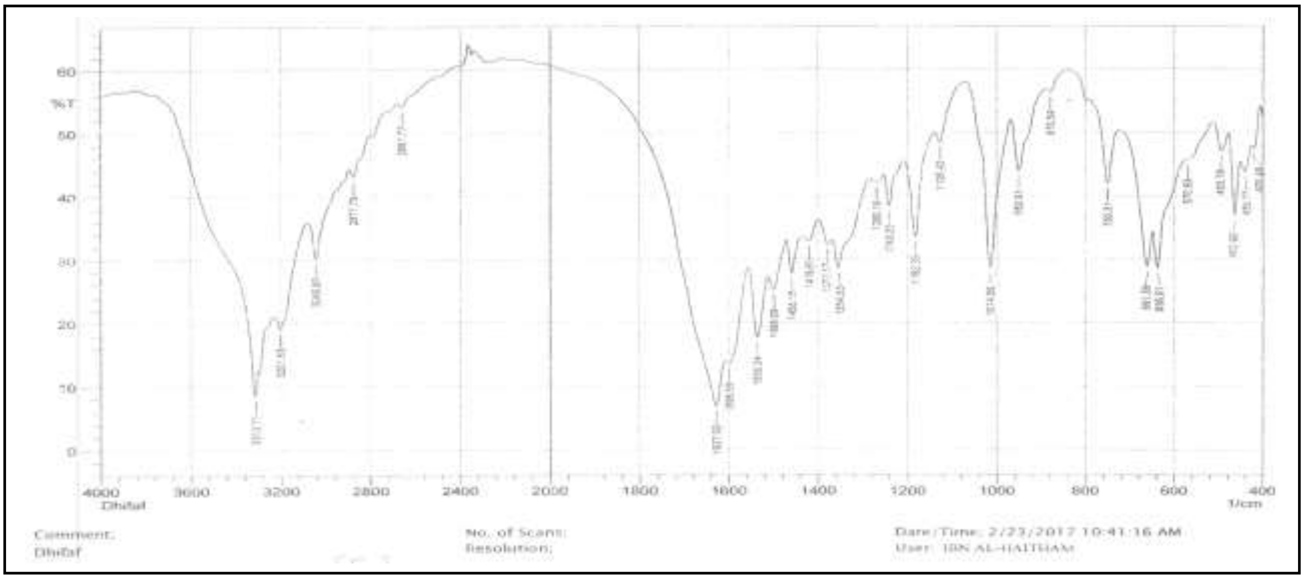


Fig.3: FT-IR band result for compound [5]

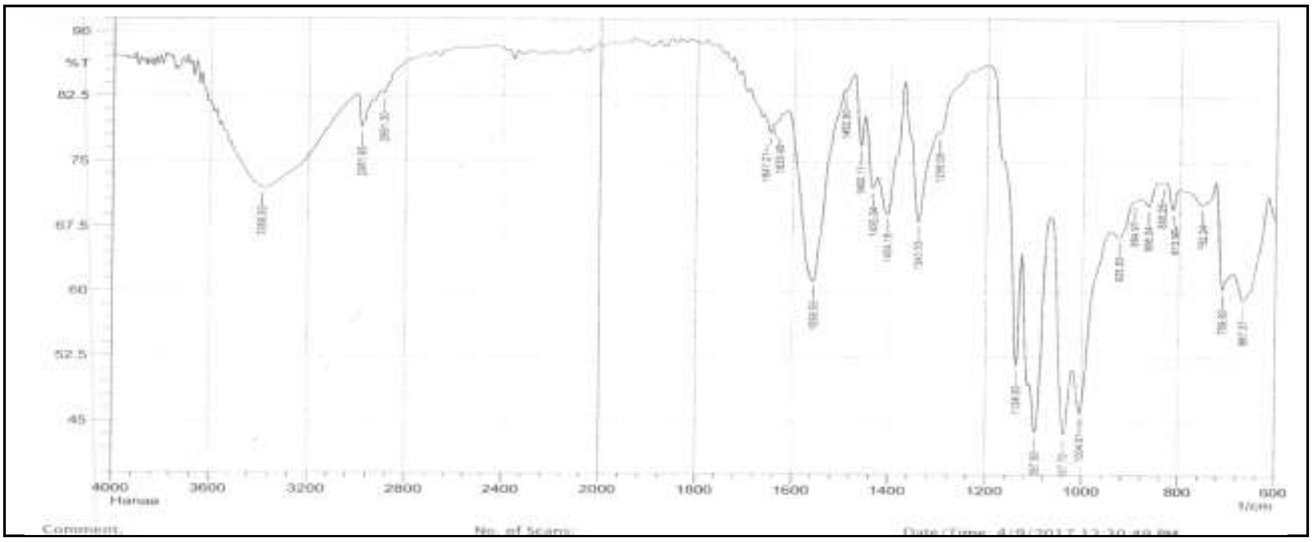


Fig.4: FT-IR band result for compound [6]

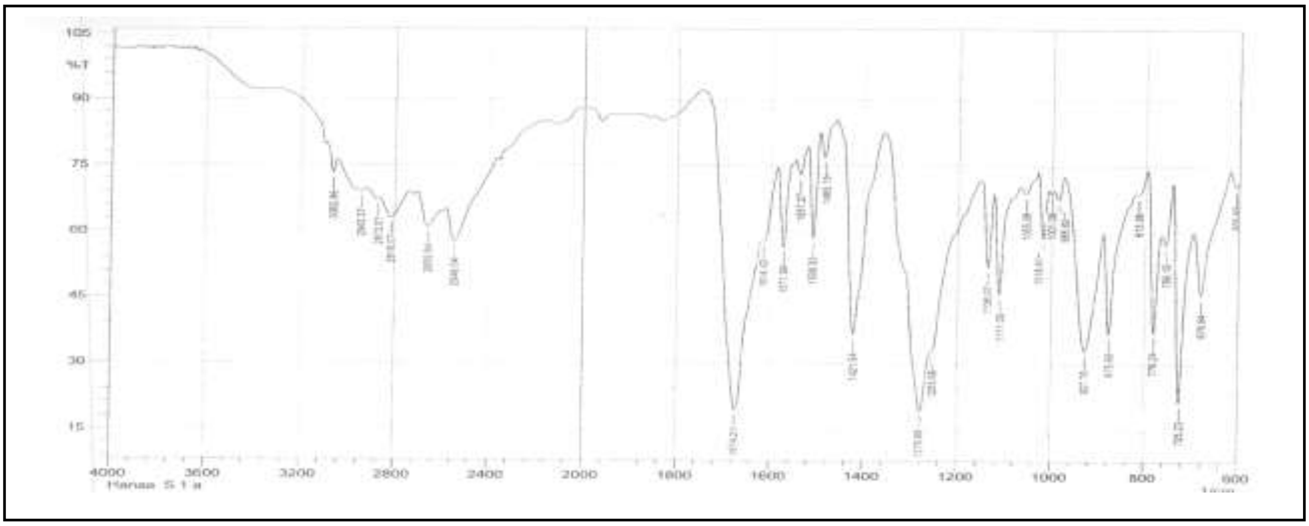


Fig. 5: FT-IR band result for compound [9]

Table 1: The IR characteristic bands for polymers [7-12]

Comp. No	(N-H)	(C-H) Ar.	(C-H) Alih.	C=O	C=N	C-N	CH_2 bend	C=C
7	-	-	2924,2854	1720	1614		1485	-
8	-	-	2922,2852	1710	1631		1435	-
10	3203	-	2924,2852	1695	-	1371	1450	-
11	3209	-	2945,2873	1662	-	1375	1485	-
12	3101	3062	2943,2814	1674	-		1421	1571

Thermal Transitions

The thermogravimetric (DSC/TG) curves of polymers have achieved for heating rate of 10 °C. min^{-1} under argon measured in temperatures within 0 to 600 °C range. Polymers [10-12] have greater Tg magnitude and these polymers are feasibly adopted with inter and intra-interactions hydrogen bonding that feasibly made among residual N-----H and other polar groups (there are another endothermic transitions can be discerneed from the thermograms. Almost all the preparing compounds showed good thermal stability and the digram contains the values of T_g and T_m of the prepared compounds.

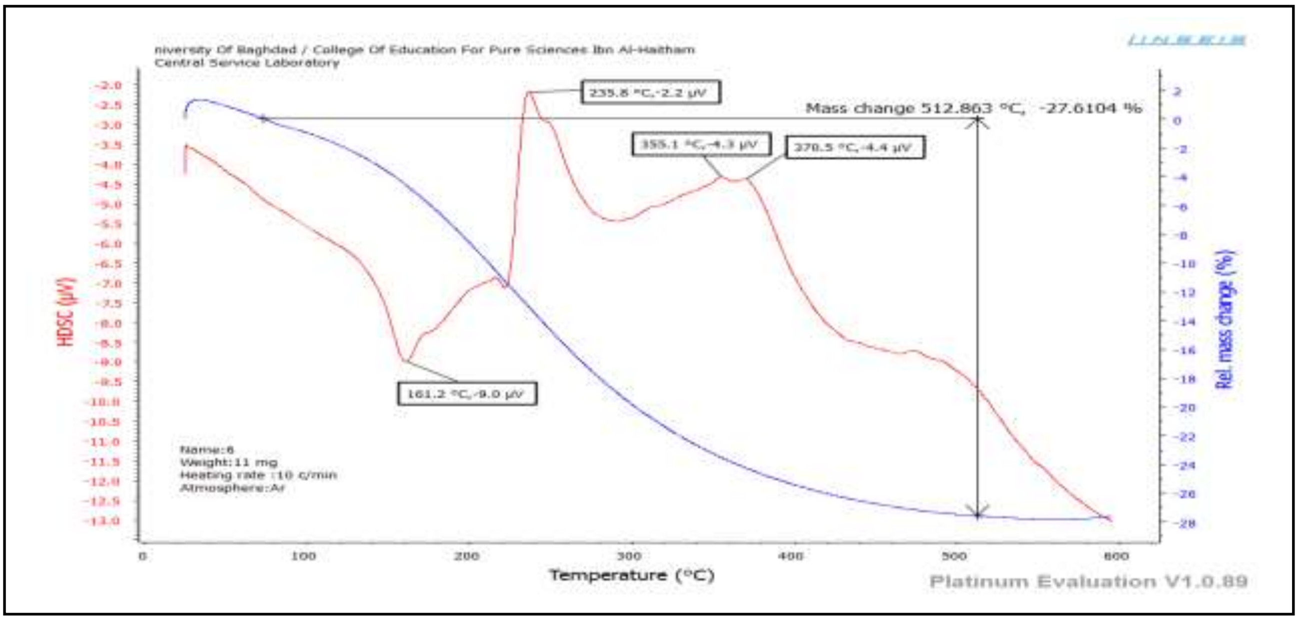


Fig6: Thermographs (TGA and DSC)of polymer [7]

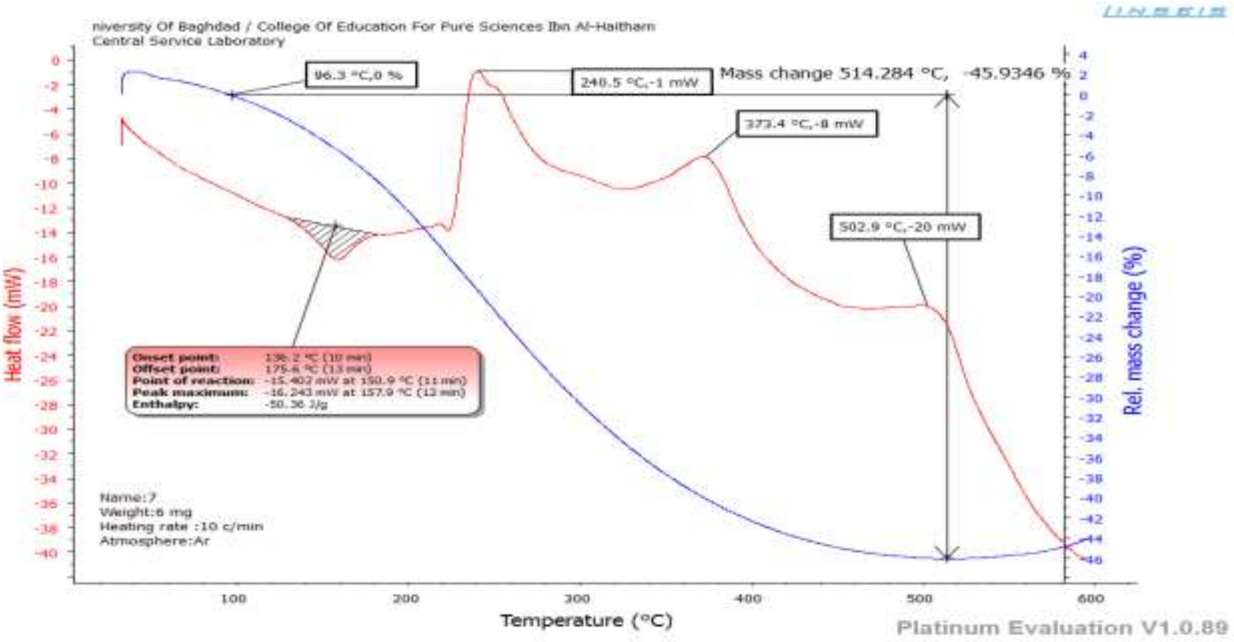


Fig7: Thermographs (TGA and DSC)of polymer [8]

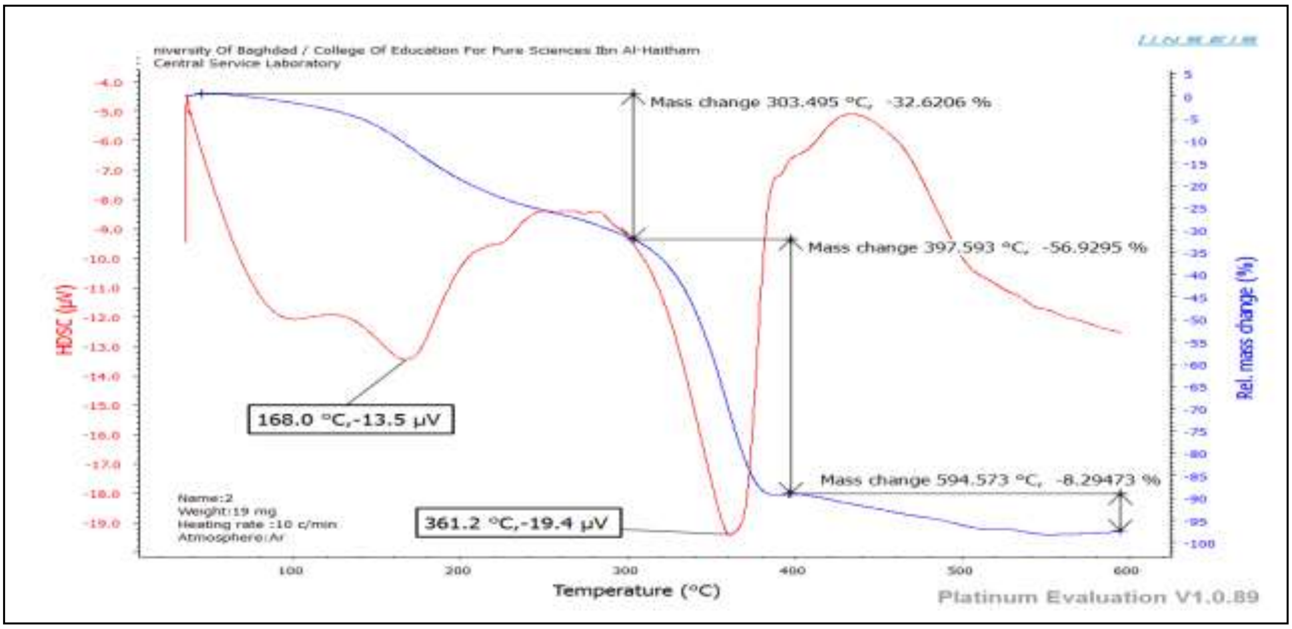


Fig8: Thermographs (TGA and DSC)of polymer [9]

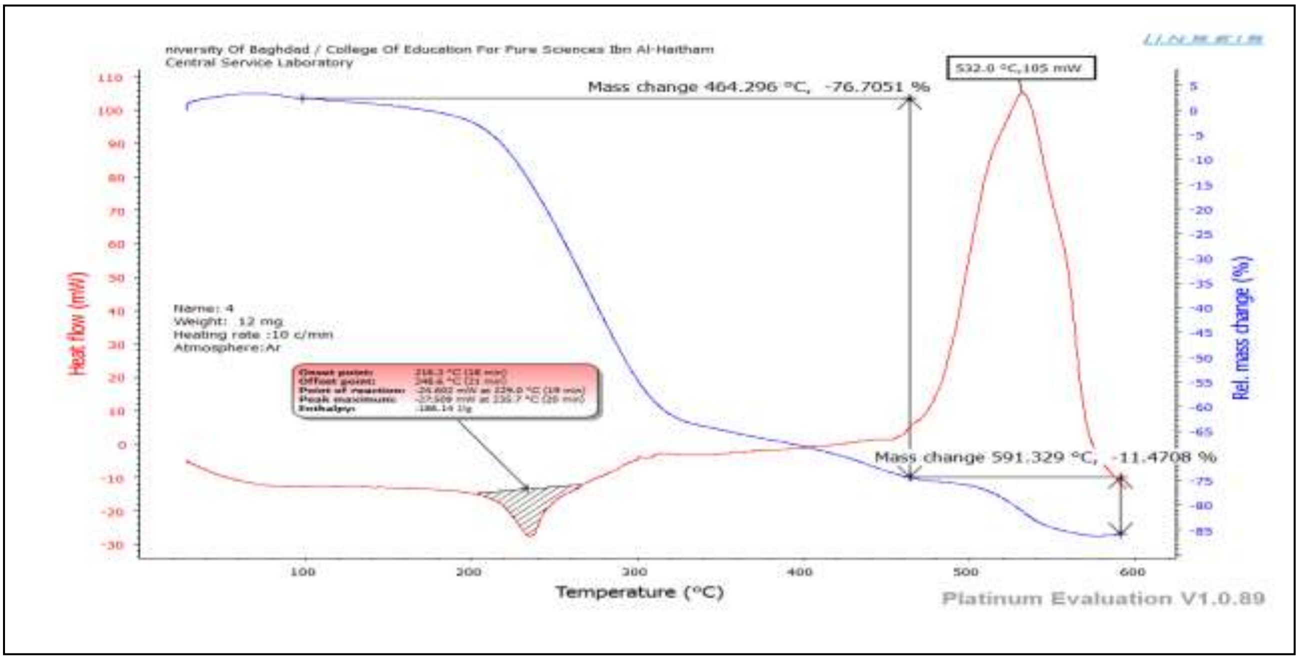


Fig 9: Thermographs (TGA and DSC)of polymer [10]

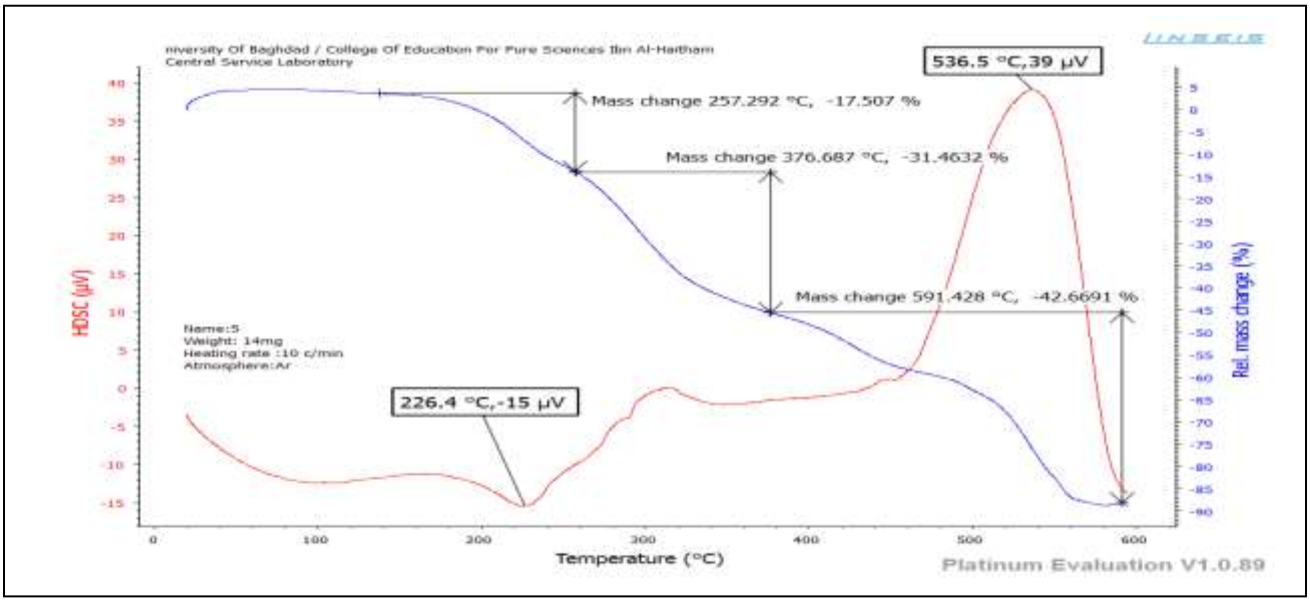


Fig 10: Thermographs (TGA and DSC)of polymer [11]

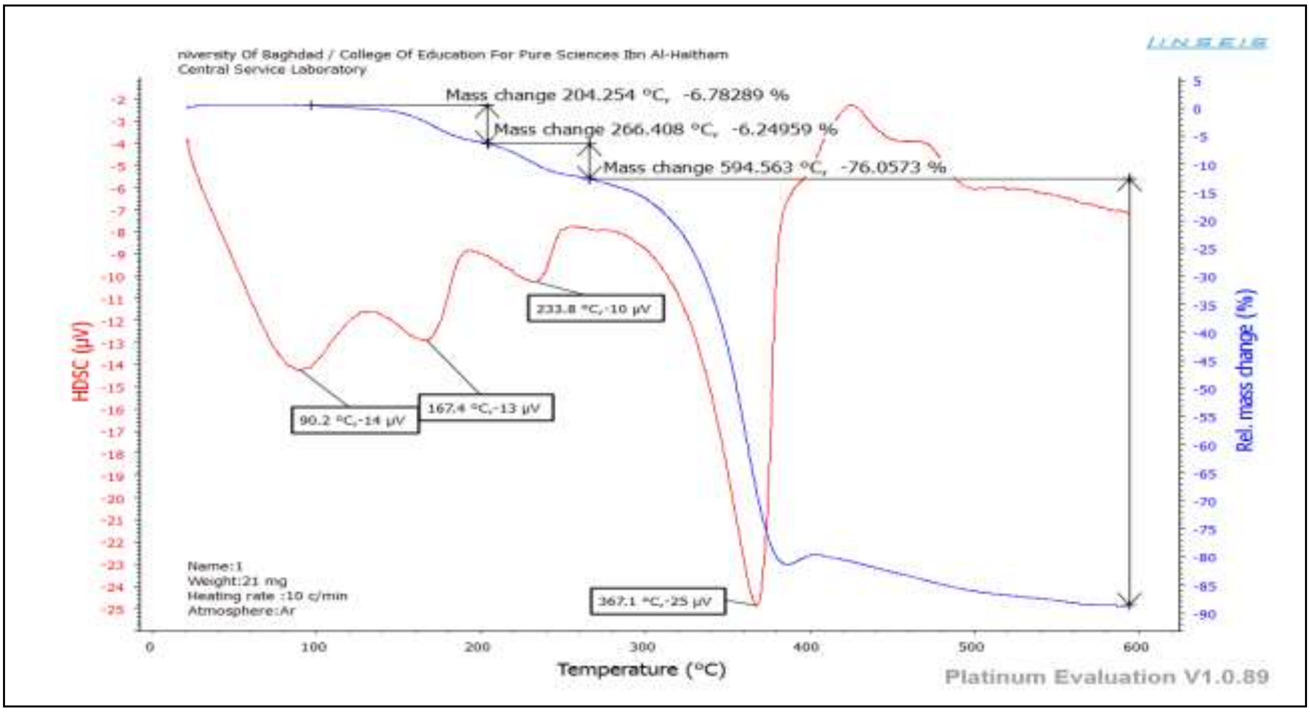
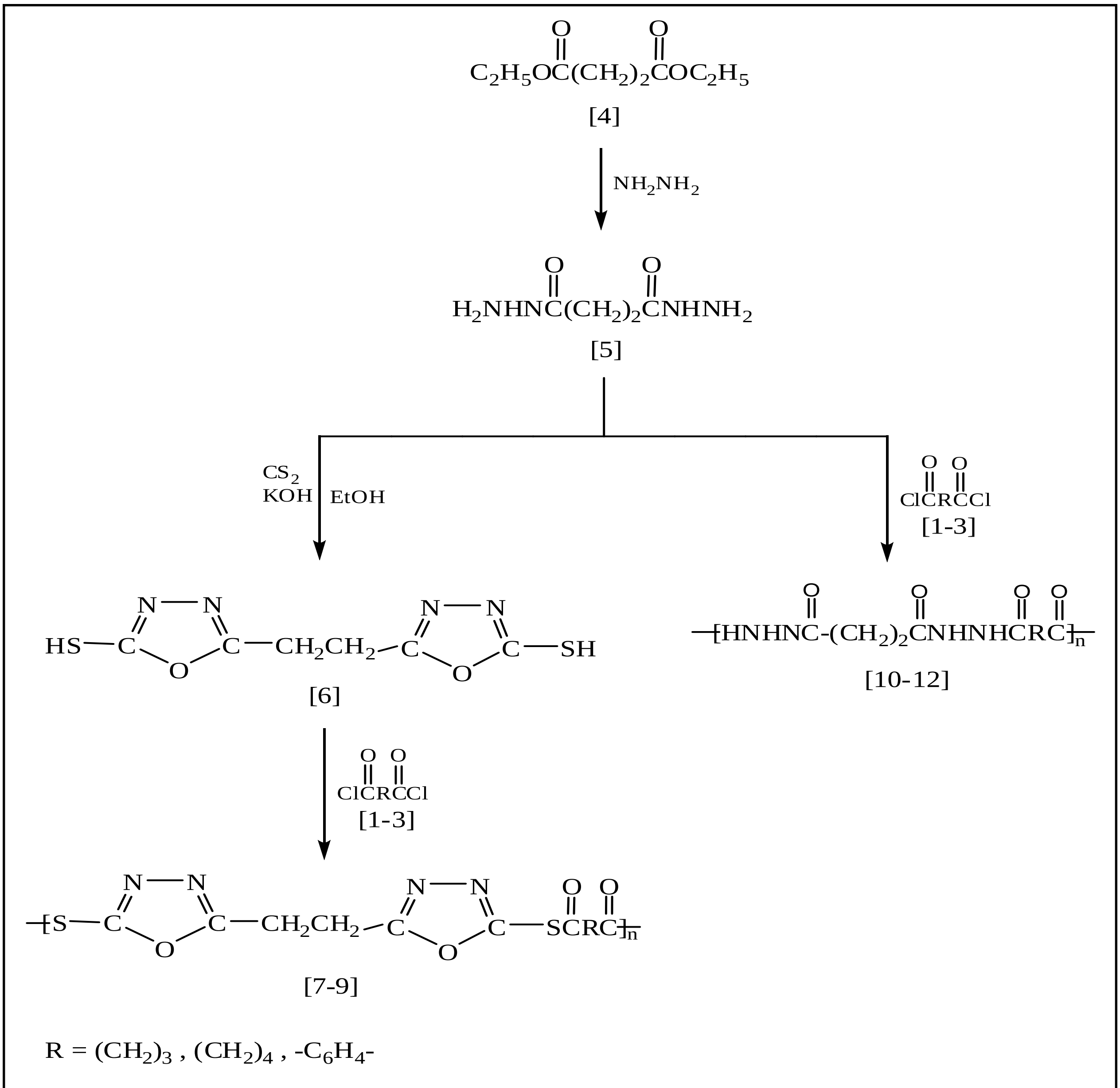


Fig11: Thermographs (TGA and DSC)of polymer [12]



Scheme (1)

In Vitro Antibacterial Activity

The antibacterial investigation in this study has achieved based on the disc diffusion technique [14]. Compounds [7-11] have been examined for their antimicrobial performance in *vitro* in contradiction of single strain of Gram negative bacteria (**E.coli**) along with single strain of Gram positive bacteria (**Staphylococcus aureus**). The preceding bacteria have been motivated in the Nutrient Growth media under 37 °C for one day. The organized agar along with Petri dishes have sterilized through autoclaving for 15 minutes at 121°C. The agar has been surface inoculated homogeneously from the broth culture of established microorganisms [15].

Table 2: Evaluation of antibacterial activity of the compounds

Compound No.	Staph.	E.Coli.
[7]	_	+
[8]	+	+
[9]	++	++
[10]	+	+
[11]	+++	+++

Key to Symbols
Extremely active = +++ (inhibition zone > 20 mm).
Medium active = ++ (inhibition zone 11-20 mm).
Marginally active = + (inhibition zone 5-10 mm).
Inactive = - (inhibition zone <5 mm.

Conclusions

Here, in this work, constructions of monemers with Oxadiazole heterocyclic rings with categorization were succeeded.

- The existence of heterocyclic rings provides greatly thermal stability as well as worthy solubility for the organized polymers.
- Results (FTIR, X-Ray diffraction, TGA and DSC) of formed monemers and polymers have been compatible with suggested structure analytically and spectrally.
- The existence of heterocyclic rings in several equipped monomers. Besides, polymers have in elevation or small organic activity.
- The organized polymers possess very melting point analogous to homopolymers.

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