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Synthesis of New Heterocyclic Polymers from Chalcone

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Abstract

Chalcone derivative were synthesized by reaction of some benzaldehyde derivatives with acetophenone or substituted. Then the products were reacted with hydrazine hydrate, semicarbazide and phenyl hydrazine, to give the heterocyclic derivatives of pyrazole to use preparation monomers from acid chloride to give polymers by free radicals. The structure of these compounds characterized by FT-IR, ^{1}H-NMR spectra, melting points, TLC, Vis, Uv/Vis and X-ray, These compounds were also screened for their antibacterial activities.

Key words: chalcone, heterocyclic, pyrazole and polymer.

Introduction

Chalcones were prepared by condensation of substituted acetophenone with aromatic aldehydes in presence of suitable condensing agent [1-3].

Chalcones undergo a variety of chemical reactions that leads to many heterocyclic compounds [4-6]. Chalcones have been used as intermediates for the preparation of compounds having therapeutic value [7-8], view of the varied biological and pharmacological applications, we have planned to synthesize some heterocyclic derivatives of chalcone and test their antibacterial activity[9].

Heterocyclic compounds are those cyclic compounds in which one or more of the ring carbons are replaced by another atom. The non-carbon atoms in such rings are referred to as hetero atoms. The most common hetero atoms are nitrogen, oxygen and sulfur, but other atoms such as boron, phosphorus, or silicon can also be members of heterocyclic rings [10]. Heterocyclic compounds have a wide range of applications, but are of particular interest in medicinal chemistry and industrial application (11).

Many heterocyclic compounds were found in natural products such as alkaloid (alkali-like), this compound is containing nitrogen atom in the ring, and for example papaverine from the opium poppy (papaver somniferous) is containing pyridine (12).

Pyrazole is one of a class of organic heterocyclic compounds containing a five member aromatic ring structure composed of two nitrogen atoms and three carbons. But pyrazoline it is a class of organic heterocyclic compounds containing a five member not aromatic ring structure composed of two nitrogen atoms and three carbons.

Variously substituted pyrazolines and their derivatives are important biological agents and a significant amount of researches for their activity has been directed towards this class. In particular they are used antitumor (13) antibacterial, antifungal, and antiviral (14-15). Substituted Pyrazole presented a wide range of biological activity: it used as inhibitors and deactivators of liver alcohol dehydrogenates antitumor, anti-inflammatory and antifungal (16).

Experimental part

-Synthesis of 1-(4-hydroxyphenyl)-3-(4-methyl) prop-2-en-1-one [E] (17)

The mixture P-hydroxy acetophenone (0.2gm, 0.014 moles) with P-methyl benzaldehyde (1.8gm, 0.011 moles) were dissolved in ethanol (25 mL), Sodium hydroxide (10%) was added slowly and the mixture stirred for 4 hrs. In ice water bath, the mixture kept stirred at room temperature overnight, and then the mixture poured into ice cold water and acidified with diluted hydrochloric acid, filtered, washed and recrystallized from ethanol, m.p (212°C), and yield (75%).

- Synthesis of 1-carboxamide 3-(4- methyl)-5-(4-hydroxyphenyl) -4, 5-dihydro-1H-pyrazol [E_a] (18)

A mixture of semicarbazide hydrochloride (1.5gm' 0.4mole) and sodium hydroxide (5%) in absolute ethanol (10ml) with chalcone (E) (0.25gm,0.05mole) was refluxed at 75°C for (15hrs). The progress of the reaction was monitored by TLC, after the completion of reaction; the reaction mixture was poured into acidic ice water. The solid precipitate was filtered off and recrystallized from ethanol, m.p (242-246°C), and yield (75%).

-Synthesis of -3(4- methyl) 5-(4-hydroxy phenyl) 4, 5-dihydro-1H-pyrazole [E_b] [19]

Chalcone (E) (0.2gm' 0.05mole) were dissolved in ethanol 95% (10ml) and (1.0gm) NaOH and refluxed with excess of ($NH_2NH_2.H_2O$) (5-10ml) for 12hrs.at 75°C. The brown precipitate formed was filtered off and recrystallized from ethyl alcohol, m.p (217-219°C), and yield (72%).

-Synthesis of 1-phenyl 3-(4-methyl) - 5-(4-hydroxy phenyl) -4, 5-dihydro-1H-pyrazole [E_c] [20]

Chalcone (E), (0.2gm' 0.07mole) were refluxed with phenyl hydrazine (0.015mol) in absolute ethanol (15 ml) at 80°C for 11hrs; the progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was poured into acidic ice water. The solid precipitate was filtered off and recrystallized from ethanol; the compound is gummy and yields (78%).

- **Synthesis of the monomer **

- 2-[1- phenyl -3-(4-methyl)-4, 5-dihydropyrazole-5-yl-(4-phenoxy carbonyl)] styrene [E_d] [21]

The compound [E_c] (0.21gm' 0.08mole), were dissolved in dry pyridine and drops of dimethyl formamide [DMF] , then (cinnamoyl chloride) added drop wise in ice water bath with continues stirring for 5hrs, then pouted the mixture setting refluxed for an hour(70-75°C) and then poured into ice cold water and acidified with diluted hydrochloric acid , m.p(190-193°C) ,yield(57%) .

- **Synthesis of the Polymer **

-Poly 2-[1- phenyl -3-(4-methyl)-4, 5-dihydropyrazole-5-yl-(4-phenoxy carbonyl)] styrene [E_e] [22]

The polymer were prepared by free radical polymerization ,compound [E_e] (0.22gm), Benzoyl peroxide (0.001gm) was added in (10ml) tetrahydrofuran, the mixture was heated with stirring at (90°C) for 3hr. The polymers were collected by precipitation in ice water, filtration, washed several times with water and dried by diethyl ether at room temperature. m.p (207°C).

- **Synthesis of the monomer **

-2[1-phenyl -3-(4-methyl) - 4, 5-dihydro-1H-pyrazole-5-yl] phenyl acrylate [E_f] [23]

The compound [E_f] (0.14gm' 0.04mole), (0.11gm' 0.04mole), were dissolved in dry pyridine and few drops of dimethyl formamide [DMF], then (acryloyl chloride) was added drop wise in ice water bath with continues stirring for 5hrs, then potted the mixture refluxed for an hour (70-75°C) and

then poured into ice cold water and acidified with diluted hydrochloric acid, resulted compound is gummy.

- **Synthesis of the Polymer \ -Poly-2[1-phenyl -3-(4-methyl) - 4, 5-dihydro-1H-pyrazole-5-yl] phenyl acrylate [E_g] (24)**

The polymer were prepared by free radical polymerization , compound [E_f] (0.19gm), Benzoyl peroxide (0.001gm) was added in (10ml) tetrahydrofuran, the mixture was heated with stirring at (90°C) for 3hr. The polymers were collected by precipitation in ice water, filtration, washed several times with water and dried by diethyl ether at room temperature, the polymer is gummy.

Results and Discussion

-Preparation and Characterization of 1-(4-hydroxyphenyl)-3-(4-methyl) prop-2-en-1-one [E]

Chalcone is an aromatic ketone that forms the central core for a variety of important biological compounds, which are known collectively as chalcones. They show antibacterial, antifungal, antitumor and anti-inflammatory properties [25]. They are also intermediates in the biosynthesis of flavonoids. Most chalcones are typically colored yellow to orange [25]. The structure of the product assignment on its melting point and spectral FT.IR, ^{1}HNMR spectroscopy, and the purity of this compound are checked by T.L.C. technique.

Series of chalcone were prepared by Claisen-Schmidt condensation of appropriate acetophenone with appropriate aromatic aldehyde. Compound (E) containing carbon-carbon double bond (C=C) and carbonyl bond (C=O) in conjugation are known as α, β-unsaturated carbonyl compounds. The prepared chalcone (E) gave a negative result this test was proved that there was no aldehyde in compound (E) because the aldehyde was converted to keto form [26]. FT-IR spectra of chalcone (E), figure (1) gave a strong band at (1647 cm^{-1}) which belongs to conjugated carbonyl group. Another strong band belongs to carbon-carbon double bond appeared at (1606cm^{-1}) [159]. It is worth mentioning that the intensity of (C=C) band is generally more than (C=O) band for most of the chalcone.

Table (1) FT-IR spectral data of compound [E]

Co No.	υ(C-H) aromatic cm^{-1}	υ(C-H) aliphatic cm^{-1}	υ(C=C) cm^{-1}	υ(C=O) cm^{-1}	υ(-OH) cm^{-1}	Other bands cm^{-1}
E	3026	2922	1606	1647	3115	υ(CH_3) 2804

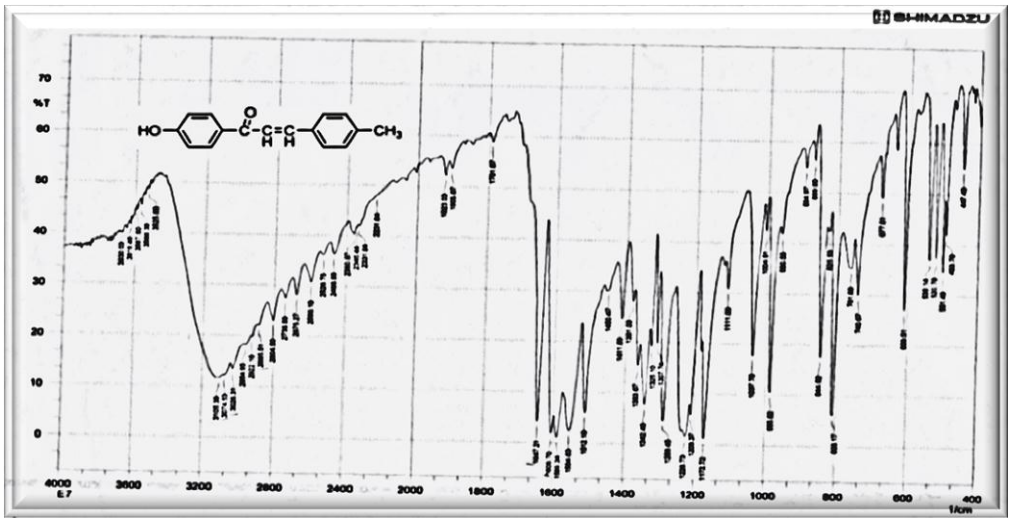


Figure (1) FT-IR spectrum of compound [E]

^{1}H-NMR spectra of compound (E), fig. (2) Shows the following characteristic chemical shifts, (2.5) ppm was due to (DMSO), the duplicate at δ (6.7, 8.8) ppm are due to the proton for the methylene group besides a signal appeared at δ (9.7) ppm (-H) of (-OH) in hydroxyl group.

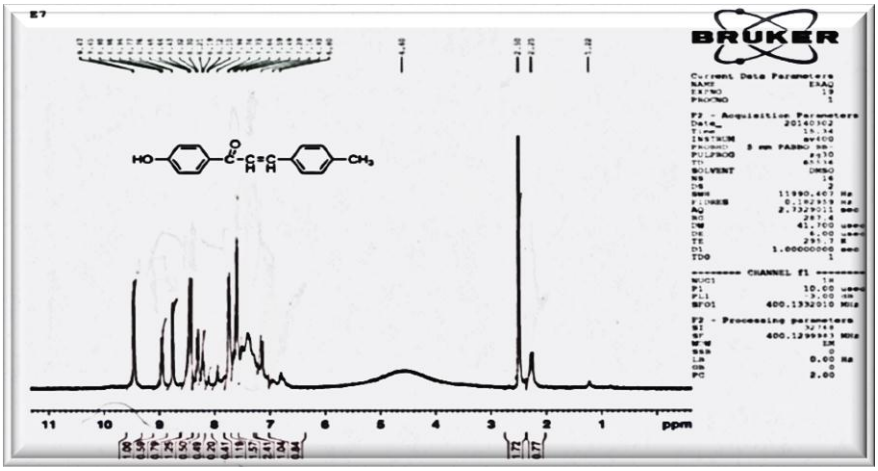


Figure (2) ^{1}HNMR spectrum of compound [E]

- **Preparation and Characterization of 1-carboxamide 3-(4-methyl)-5-(4-hydroxyphenyl) -4, 5-dihydro-1H-pyrazol [E_a]**

The condensation reaction of chalcone (E) with semicarbazide hydrochloride in ethanol absolute give compounds (Ea).

The FT-IR spectra figure(3) of compound (Ea) indicate the absence of a strong characteristic absorption band at about (1606 cm^{-1}) corresponding to olefinic double bond of chalcone (E) which disappearance of a strong absorption of carbonyl group in the chalcone (E),the amide carbonyl gives a strong absorption band at(1645cm^{-1}), appearance of a band at (3620) cm^{-1}

for (-OH)group is good evidence for the structure given to these compound [18] , FT-IR spectral data of these compound are listed in table (2).

Table (2) FT-IR spectral data of compound [E_a]

Cor No.	υ(C-H aromatic cm^{-1}	υ(C-H aliphatic cm^{-1}	υ(C=N) cm^{-1}	υ(C=O) cm^{-1}	υ(-NH_2) cm^{-1}	Other bands cm^{-1}
E_a	2962	2845	16	1	31	υ(C-N) 1265

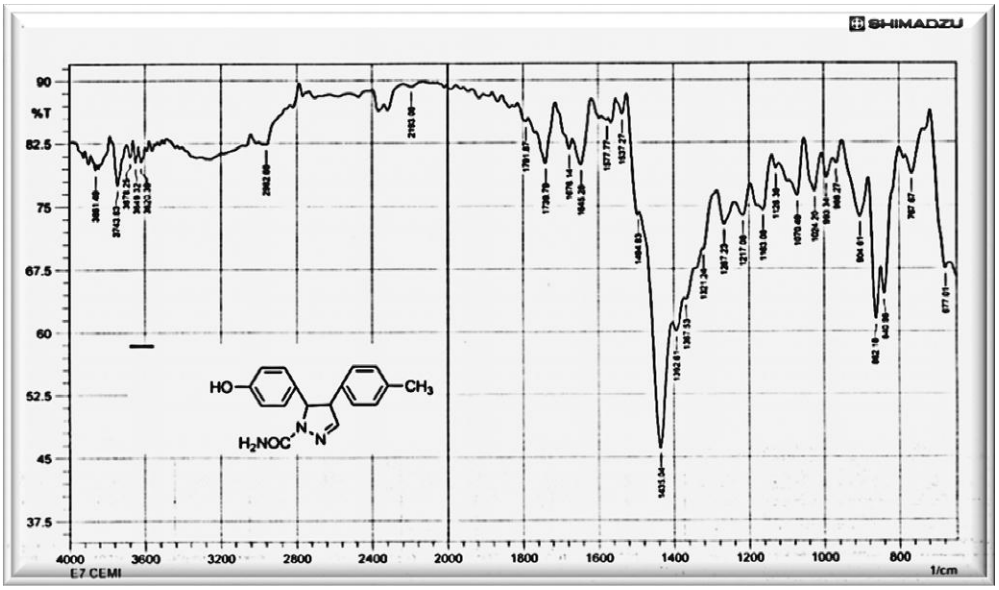


Figure (3) FT-IR spectrum of compound [E_a]

-Preparation & Characterization of -3(4- methyl) 5-(4-hydroxy phenyl) 4, 5-dihydro-1H-pyrazole [E_b]

The titled compound (E_b) was synthesized by the condensation reaction of chalcone (E) with hydrazine hydrate (*$NH_2NH_2.H_2O$*) in ethanol absolute. The FT-IR spectra for compound (E_b) as a representative model shows the disappearance of the characteristic absorption band at about (1606)$cm^{-1}$of the olefinic (C=C) which is present in the FT-IR spectrum of chalcone (E), also disappearance of (1647) cm^{-1} which attributed to the carbonyl group in the chalcone compound, and appearance of(C=N) band at (1654), (C-N) band is identified as a medium band at (1303) cm^{-1}, and absorption band at (3278) cm^{-1} is due to the (N-H) stretching [27], addition to the appearance of bands in the region (3344) cm-1 due to ν(-OH)group. Figure (4).

Table (3) FT-IR spectral data of compound [E_b]

Comp No.	υ(C-H) aromatic cm^{-1}	υ(C-H) aliphatic cm^{-1}	υ(C=C) cm^{-1}	υ(C=N) cm^{-1}	υ(N-H) cm^{-1}	Other-bands cm^{-1}
E_b	3014	2954	1571	1654	3278	υ(C-N) 1303

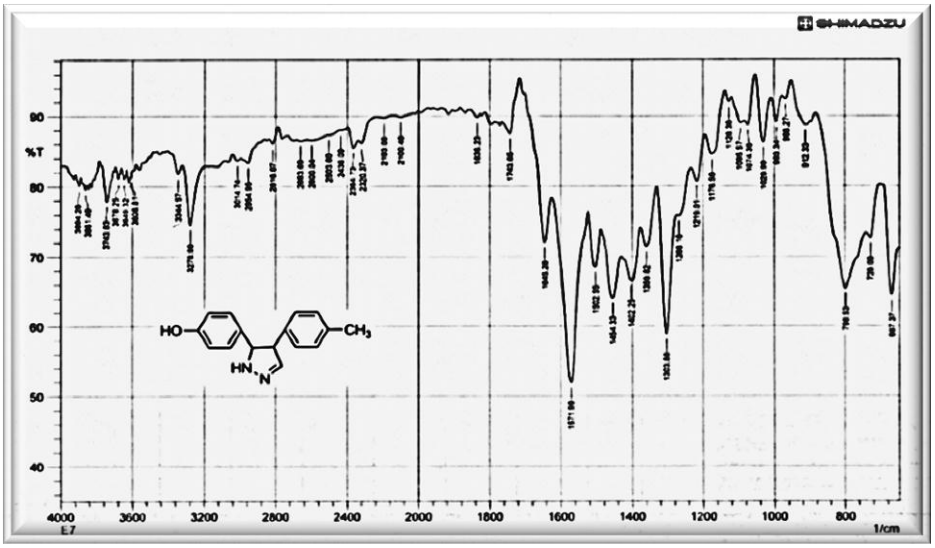


Figure (4) FT-IR spectrum of compound [E_b]

-Preparation and characterization of -1-phenyl -3-(4-methyl) 5-(4-hydroxy phenyl) -4, 5-dihydro-1H-pyrazole [E_c]

The condensation reaction of phenyl hydrazine with chalcone derivatives (E) afforded the corresponding compound (E_c) [28]. FT-IR spectral data for these compound (E_c) figure (5) the appearance of absorption bands at (1654) cm^{-1} is attributed to the (C=N) of pyrazoline ring. Absorption at (1597) cm^{-1} due to the aromatic system (C=C).

Table (4) FT-IR spectral data of compound [E_c]

Comp	υ(C-H) aliphatic cm^{-1}	υ(C-H) aromatic cm^{-1}	υ(C=N) cm^{-1}	υ(C-N) cm^{-1}	υ(C=C) cm^{-1}	Other bands cm^{-1}
E_c	2922	3026	1654	1265	1597	υ(-OH) 3298

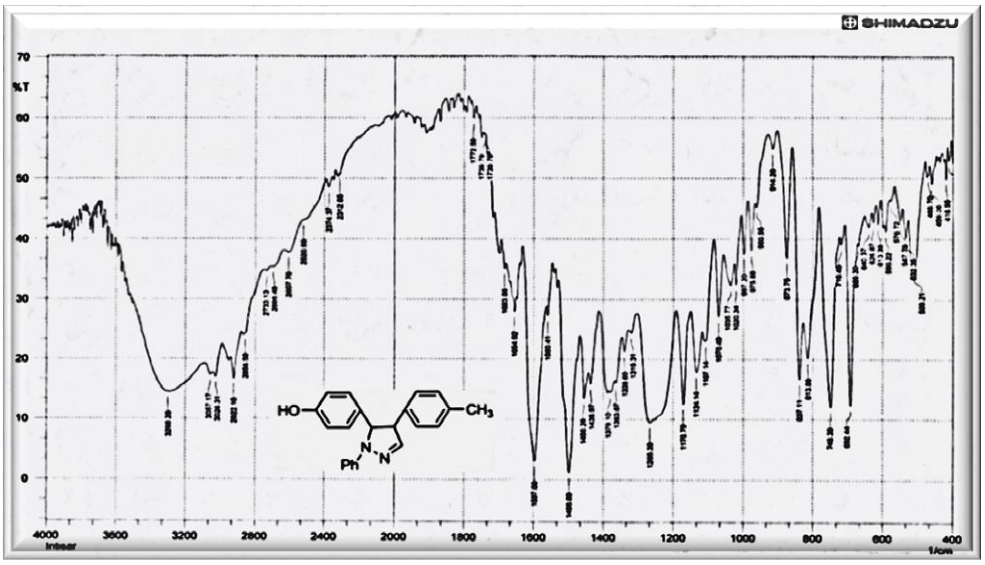
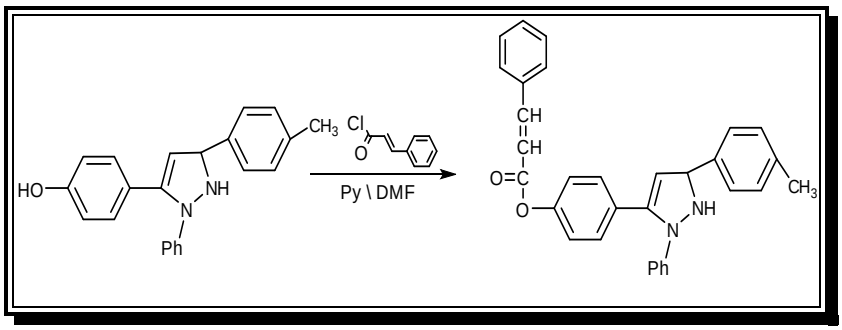


Figure (5) FT-IR spectrum of compound [E_c]

- Preparation and characterization of - 2-[1- phenyl -3-(4-methyl)-4,5-dihydropyrazole-5-yl-(4-phenoxy carbonyl)] styrene [E_d]

Monomer was prepared from the dissolved compound (E_c) in pyridine and reaction with cinnamoyl chloride [29, 30] as in the following equation.



The FT-IR spectrum for compound (E_d) as a representative model shows the appearance of the characteristic absorption band at about (1716) cm^{-1} of the carbonyl group (C=O). Also appearance of (C-N) band is identified as a medium band at (1261) cm^{-1}, and absorption band at (1168) cm^{-1} is due to the (C-O) stretching, Figures (6).

Table (5) FT-IR spectral data of compound [E_d]

Comp No.	υ(C-H) aliphatic cm^{-1}	υ(C-H) aromatic cm^{-1}	υ(C=O) cm^{-1}	υ(C-N) cm^{-1}	υ(C=C) cm^{-1}	Other bands cm^{-1}
E_d	2914	3052	1716	1261	1551	υ(C-O) 1168

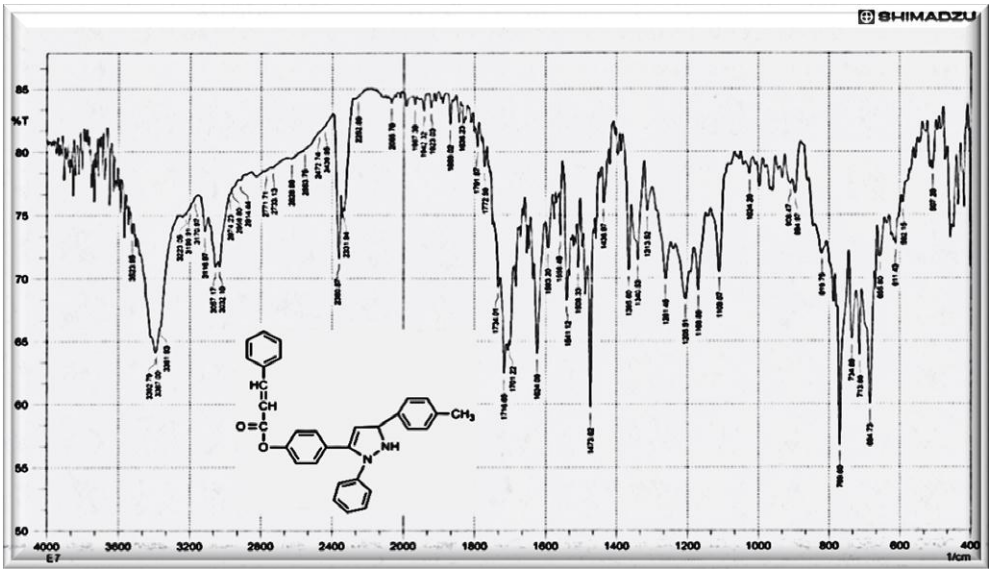


Figure (6) FT-IR spectrum of compound [E_d]

^{1}H-NMR spectra of compound (E_d), fig. (7) shows the following characteristic , the multiple at δ (6.62-8.03) ppm are due to the proton for the methylene group.

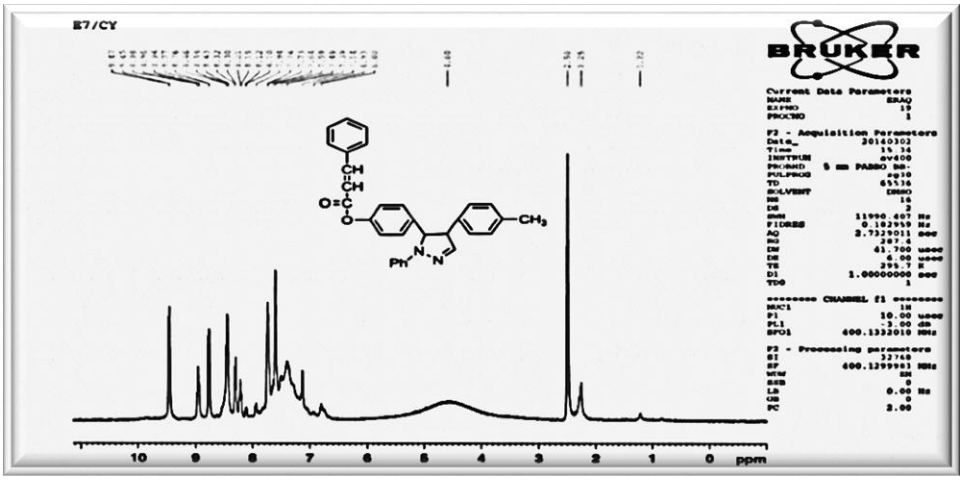


Figure (7) ^{1}HNMR spectrum of compound [E_d]

- Preparation and characterization of poly 2-[1- phenyl -3-(4-methyl phenyl)-4, 5-dihydropyrazole-5-yl-(4-phenoxy carbonyl)] styrene [E_e] (22)

The prepared polymer was characterized by FT.IR and UV/Vis spectroscopy. The FT.IR spectrum of compound [E_e] fig. (8) Showed the following bands at (1074) cm^{-1} due to υ (C-O), at (2922, 2953) cm^{-1} for symmetric and asymmetric stretching vibration of (-CH) aliphatic, at (1759) cm^{-1} for υ (C=O).

Table (6) FT-IR spectral data of compound [Ee]

Comp No.	υ(C-H) aliphatic cm^{-1}	υ(C-H) aromatic cm^{-1}	υ(C=C) cm^{-1}	υ(C-N) cm^{-1}	υ(C=O) cm^{-1}	Other bands cm^{-1}
E_e	2953 2922	3062	1598	1226	1759	υ(C-O) 1074

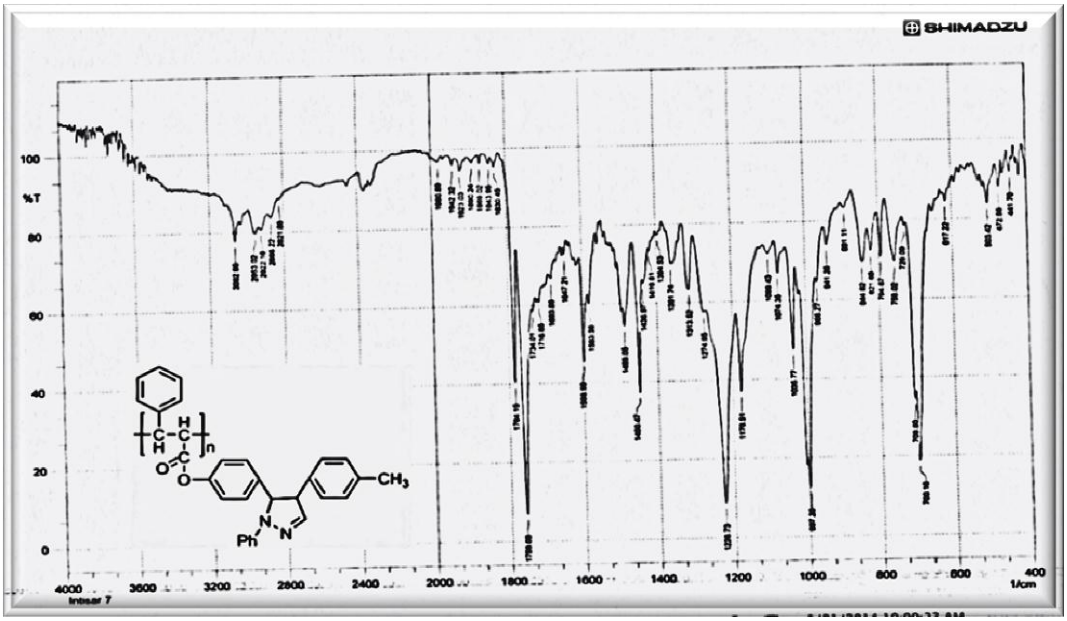
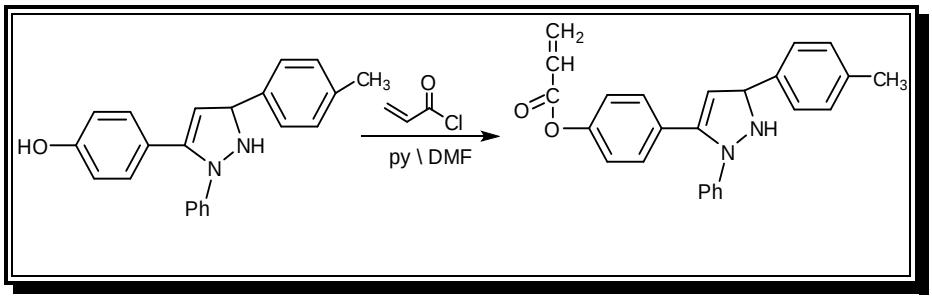


Figure (8) FT-IR spectrum of compound [E_e]

-Preparation and characterization of -2[1-phenyl -3-(4-dimethyl amino phenyl) - 4, 5-dihydro-1H-pyrazole-5-yl]phenylacrylate [E_f]

Monomer prepared from dissolved compound (E_f) in pyridine and the product reacts with acryloyl chloride [23, 31] as in the following equation.



The FT-IR spectrum for compound (E_f) as a representative model shows the appearance of the characteristic absorption band at about (1734) cm^{-1} of the ester (C=O) which is not present in the FT-IR spectrum of (Ee). Also appearance of (1601) cm^{-1} which attributed to the (C=C) in the acryloyl compound, (C-N) band is identified as a medium band at (1255) cm^{-1}.

Table (7) FT-IR spectral data of compound [E_f]

Comp No.	υ(C-H) aromatic cm^{-1}	υ(C-H) aliphatic cm^{-1}	υ(C=O) cm^{-1}	υ(C=C) cm^{-1}	υ N) cm^{-1}	Other bands cm^{-1}
E_f	3058	2926	1732	1601	1233	υ(C-O) 1176

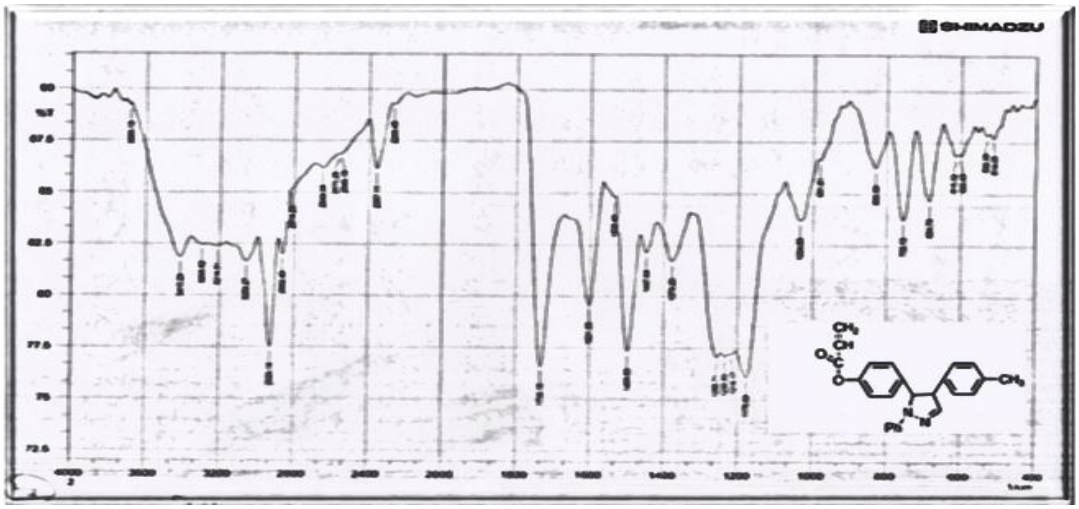


Figure (9) FT-IR spectrum of compound [E_f]

- Preparation and characterization of Poly-2[1-phenyl -3-(4-methyl phenyl) - 4, 5-dihydro-1H-pyrazole-5-yl]phenylacrylate [E_g][(24)]

The prepared polymer were characterized by FT.IR and UV/Vis spectroscopy, The FT.IR spectrum of compound [E_g], fig.(10) showed the following bands at (1062)cm^{-1} due to υ (C-O), at (2958,2926)cm^{-1} for symmetric and asymmetric stretching vibration of (CH) aliphatic, at (1734) cm^{-1} for υ (C=O).

Table (8) FT-IR spectral data of compound [E_g]

Comp No.	υ(C-H) aliphatic cm^{-1}	υ(C-H) aromatic cm^{-1}	υ(C=O) cm^{-1}	υ(C-N) cm^{-1}	υ(C=C) cm^{-1}	Other-bands cm^{-1}
E_g	2926 2958	3064	1734	1261	1600	υ(C-O) 1026

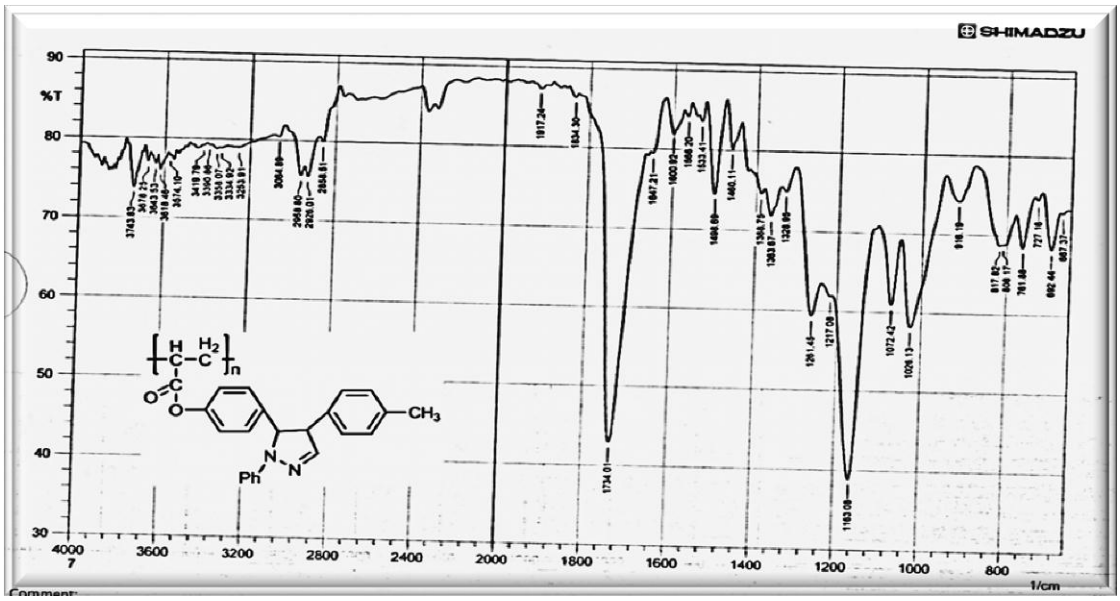


Figure (10) FT-IR spectrum of compound [E_g]

Table (9) UV \Vis spectrum of prepared polymers

Polymer No.	**Wavelength**	**Description**
E_e	*286 , 322 nm*	due to(π → π*) and (n- π*) transitions
E_g	*246 ,272 nm*	due to(π → π*) and (n- π*) transitions

Physical Properties

Comp No.	***Molecular Formula***	***Molecular Weight***	***Yield %***	***M.P***	***Rf***	***Color***	***Recryst Solvent***
E	$C_{16}H_{14}O_2$	238.28	75	212	0.83	Yellow	Ethanol
E_a	$C_{17}H_{17}N_3O_2$	295.34	75	242-246	0.88	White earthy	Ethanol
E_b	$C_{16}H_{16}N_2O$	252.31	87	225-228	0.88	Dark brown	Ethanol
E_c	$C_{22}H_{20}N_2O$	328.41	80	256 Dec	0.88	Light yellow	Ethanol
E_d	$C_{31}H_{26}N_2O_2$	458.55	78	148-150	0.86	auburn brown	Ethanol
E_f	$C_{25}H_{22}N_2O_2$	382.45	70	157-160	0.88	Light red	Ethanol

Microbiological tests \

In this work, the antibacterial test was performed according to the disc diffusion method. Heterocyclic rings are considered an important class of compounds having a wide spectrum of biological activity, the heterocyclic compounds are well known for their antibacterial and antifungal activities [32]. The results of the preliminary screening tests are listed in Table (10).

Table (10) Biological activities of the synthesized compounds

Comp No.	***E.Coli***	***Staphylococcus***	***Pseudomonas***	***Bacillus***	***E.Coli & Tetracycline***	***Staphylococcus & Vancomycin***
E	14	12	15	-	16	13
E_a	-	10	16	13	14	14
E_c	14	11	4	-	11	13
E_d	12	11	24	8	11	13
E_e	14	21	12	12	24	22
E_f	11	13	11	11	15	21
E_g	13	16	11	15	21	24

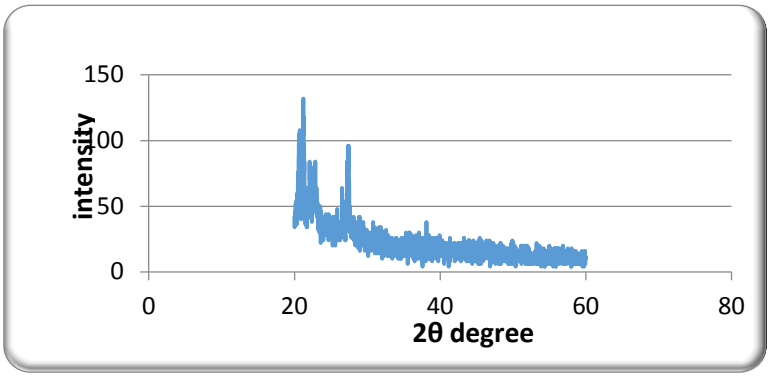


Figure (11) X-Ray of polymer [Ee]

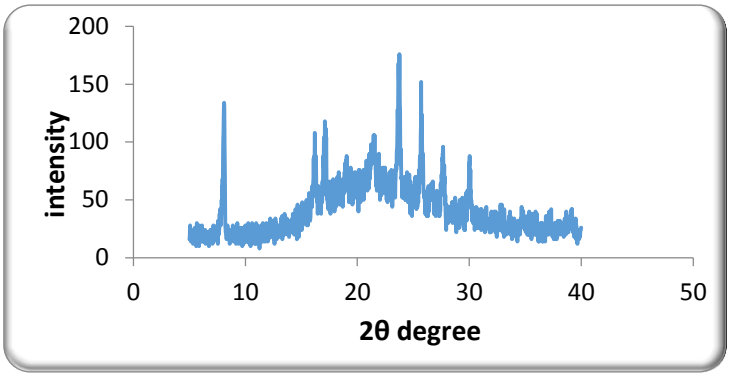


Figure (12) X-Ray of polymer [Eg]

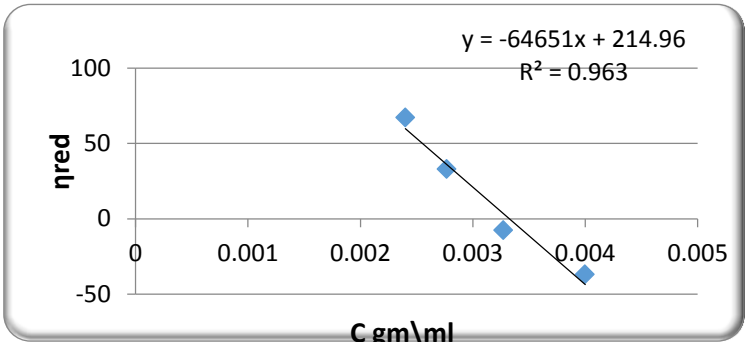
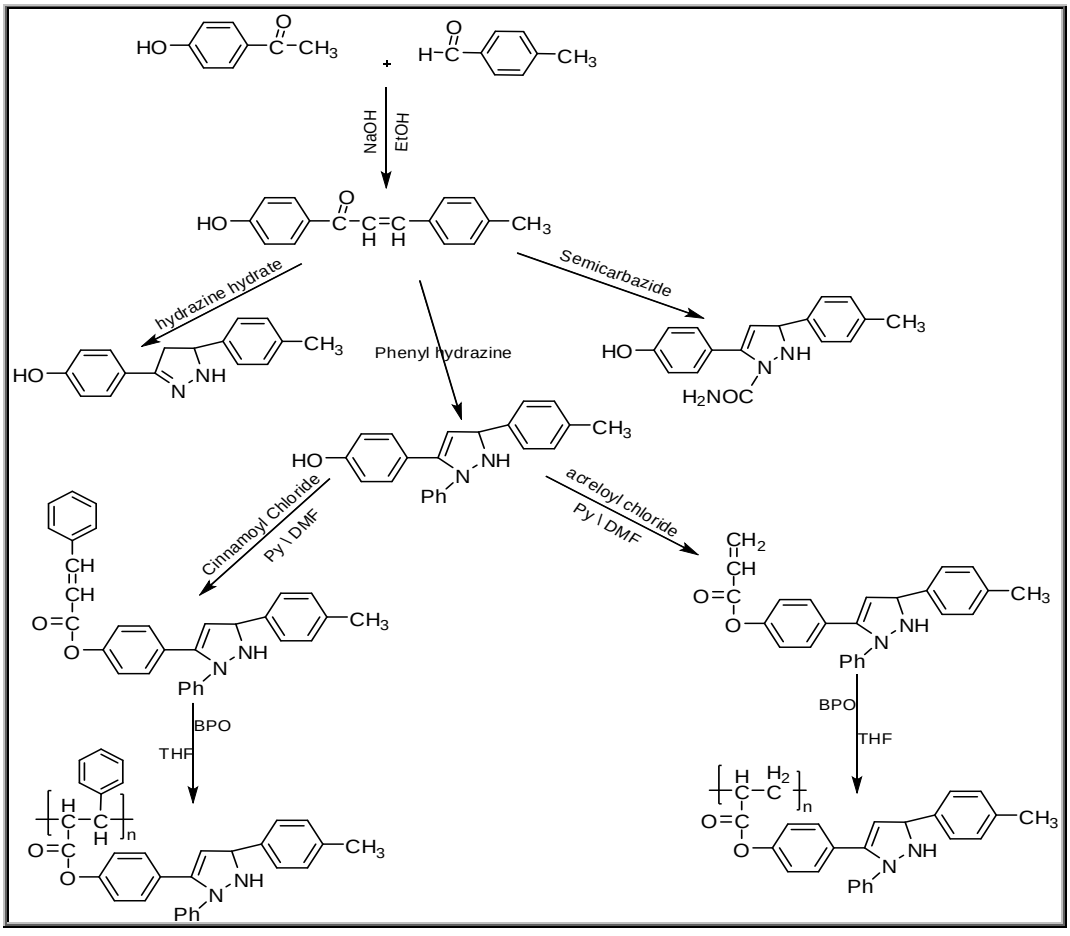


Figure (13) intensity viscosity of polymer [E_e]



Conclusion

Several points are summarized as the following:

1-It has been found we succeeded to building heterocyclic compounds as monomers to be polymerized.

2- The spectral data (FT.IR and ^{1}HNMR) of synthesis monomers were in full agreement with the proposed structure.

3- Biological activity of prepared polymers was studied and exhibited an inhibition effects, biological activity against *E.Coli& Tetracycline and Staphenococcucs& Vawcomycin.*

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