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Synthesizing and Characterizing Some Pharmaceutical Resins Using Mannich Bases and Studying Their Biological Activity

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Abstract

Mannich bases functional groups were used, which is considered an inventive phenol formaldehyde resin was created in this study. The project was divided into two phases. The first portion involves creating a monomer of Mannich bases through a condensation reaction. The reaction occurs between aromatic aldehyde (P-hydroxy benzaldehyde) and primary amines like (Metoclopramide, trimethoprim, 2-aminobenzothiazoly, sulfamethoxazole, and 4-amino antipyrine) and with minor amines such as Carbazol. By condensing Mannich bases made in part one with formaldehyde and phenol, the second stage included making phenol formaldehyde resin with Mannich bases. The synthesized resins (Phenol-formaldehyde) have been characterized. The characterization was done by (1H-NMR, and FT-IR) to determine the degree of biological activity associated with some of the active groups.Keywords: Carbazole, Mannich bases, phenol formaldehyde, polymerization and Resin.

Keywords: Biological activity, Carbazol, Mannich bases, Pharmaceutical Resins, Phenol formaldehyde.

Introduction

Phenol formaldehyde (P.F.) resin is considered one of the extremely active resins, it can be gained from reacting phenol with formaldehyde [1]. This resin, and resin-adhesives owns several industrial applications, particularly in the production of important materials like plastics[2]. Phenol formaldehyde resin mostly display high thermal stability, and high-mechanical stability, as well as to whether stability. Thus, (P.F.) resins are helpful for using them in imbibition and adhesives is constrained by slow treating rate and rising necessary curing temperature compared to other thermosetting adhesives [3]. A novel sequence of phenol formaldehyde resin was organized in this work by reacting part of organic compound phenol, as well as to other organic compound formaldehyde to Mannich bases. The current type of reactions has a great significance due to their wide usages in pharmaceutical chemistry preparation and interaction[4]. They were intensively studied mostly due to their applications in synthesis several compound especially organic materials particularly for manufacturing pharmaceutical, cosmetically, dyes, anti-bacterial , anti-cancer, anti-inflammatory, in addition to anti-convulsant [5]. Hence amine exactly secondary amine was employed in prepare bases like Mannich bases is carbazole. Carbazole derivatives are famous for their pharmacological activities[6]. It is obvious from the literature that the derivatives of carbazole moiety enjoy inclusive spectrum of pharmacological activities, such as antibacterial[7], antitumor[8], antifungal[9], antioxidant antineo plastic, and larvicrdal activity. Also carbazole along with its derivatives are a vital type of nitrogen inclosing aromatic heterocyclic compounds, own required electronic and charge transport characteristics, along with large π- conjugated system, and the numerous

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functional groups are simply presented into the structurally rigid carbazole ring[10].

Materials and Methods

The chemicals used, in the current study, were characterized in the Table 1 below:

Table 1. Materials and their description

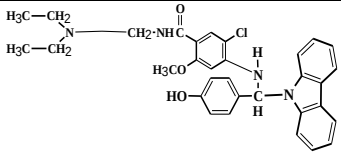
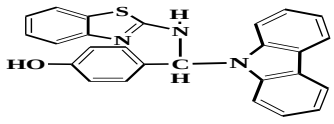
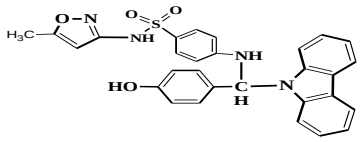
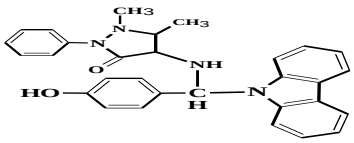
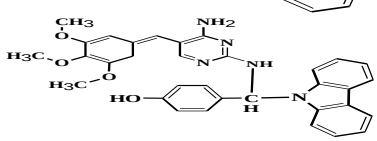
	Materials	Description
1	Chemicals	Some materials are purchased from Aldrich ND Fluka, others from Sigma, other parts were purchased from DDH, and Merch. All chemicals were not purified.
2	Melting Point Apparatus Stuart Scientific SMP3.	Used to record the uncorrected melting-points apparatus.
3	SHIMADZU FT-IR-8400	For Registering [(FT-IR)] spectrum
4	FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FTIR)	Employed to attain an infrared-spectrum of absorption with 4000-6000 cm^{-1} spectral range.
5	Bruker [(500 MHz) instrument	For Consequently (1H-NMR) spectra determination using (DMSO-d6) and (TMS)

At the Al-albayt University in Jordan's Department of Chemistry, internal reference measurements were taken.

The Mannich Base Synthesis Procedure [1,2]

Using round bottom flask, Carbazole (0.001 mol) Solution was mixed with a ten ml of Dimethyl-formamide. At the same time 0.002 mol of various amins like [(metoclopromide,,2-aminobenzothiothiazoly, sulfamethaxazole, 4-amino antipyrene, and trimethoprim)] and [p-hydroxy benzaldehyde (0.002mol)] were added and then refluxed for five hours. The utilized solvent was evaporated, and the resulting residue was decanted into ice water while being constantly agitated. After being filtered, the precipitate from the DMSO was re-crystallized. Table 2 provides a list of the physical characteristics of the items (A-E).

Table 2. Physical properties of prepared Mannich bases (A-E)

Comp.No.	Molecular Structure	Color	Yield %	FW	M. P. C°	Molecular weight
A		Brown	60	$C_{33}H_{35}ClN_4O_3$	147-149	313.82
B		Yellow	70	$C_{26}H_{19}N_3OS$	165-167	150.2
C		Light brown	76	$C_{10}H_{11}N_3O_3S$	148-150	253.27
D		white	65	$C_{30}H_{28}N_4O_2$	175-177	205.25
E		Light brown	40	$C_{33}H_{31}N_5O_4$	152-155	292.29

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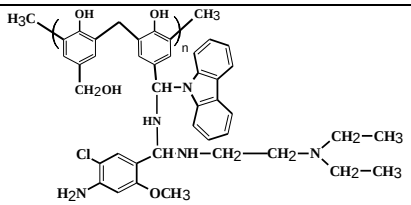
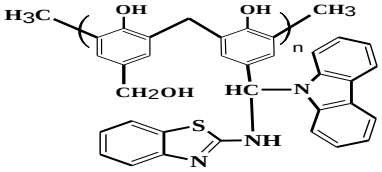
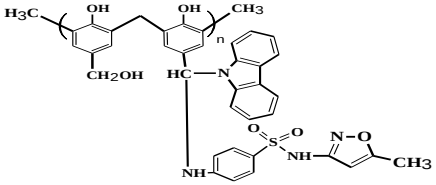
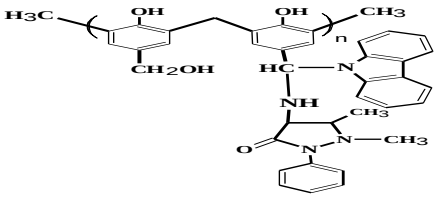
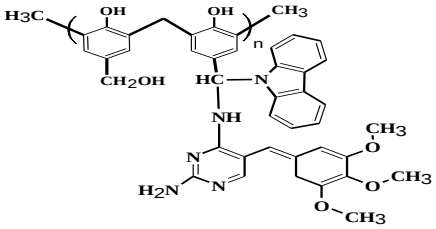
Phenol-Formaldehyde Synthesis: General Procedures.[2]

Through batch polymerization using a 6% catalyst, phenol-formaldehyde resin was created using 1to 2 as molar ratio for phenol, formaldehyde. While catalyst started in activating the reaction, phenol and formaldehyde were combined in a flask to create Mannich bases. Once the combination reached a temperature of 70 °C, it was heated to 90 °C to recover the lost heat, and then the same process was used to produce it in 93–95 °C for an hour. Table 3 shows the physical characteristics of resin (Aa-Ee).

Table 3. Physical properties data of prepared polymers (Aa-Ee)

Polymer No.	Polymer Structure	Color	Yield %	Molecular weight for repeated unit	Softing Point C^0	Molecular formula for repeated unit
Aa		Dark gray	80	706.34	215-218	$C_{41}H_{44}ClN_5O_4$
Bb		Pale red	85	527.66	190-192	$C_{34}H_{25}N_3O_3S$
Cc		Red	80	658.74	175-179	$C_{37}H_{30}N_4O_6S$
Dd		Dark Brown	75	610.72	205-208	$C_{38}H_{34}N_4O_4$
Ee		Brown	70	695.29	185-188	$C_{41}H_{37}N_5O_6$

Results and Discussion

The current research focused on functional groups that had been generated from the synthesis of [phenol formaldehyde resin] which was modified by Mannich bases Eq. 1.

This project was divided into several sections. In section one, new Mannich bases were created through the reaction of caebazole, parahydroxybenzaldehyde, and various primary amines (1–5), and their antifungal properties were tested.

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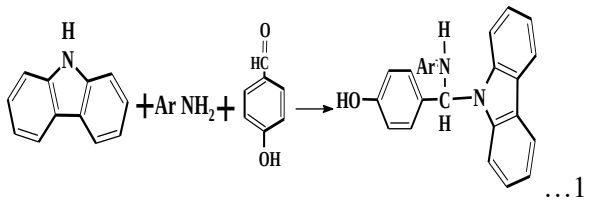

…1

Using appropriate spectral methods (1H-NMR, FT-IR), the active groups and structures of freshly produced materials were characterized and confirmed.

The results of [FT-IR spectrum for compound] (B) presented in Fig. 1 showed an absorption band at 3105 cm^{-1} that belonged to {the (N-H) amide}, as well as bands at 3051, 2812, 1643, and 3475 cm^{-1} that were caused, respectively, by [the (C-H) aromatic, (C-H) aliphatic, [the (C=N)], and [the (O-H)] of phenol.

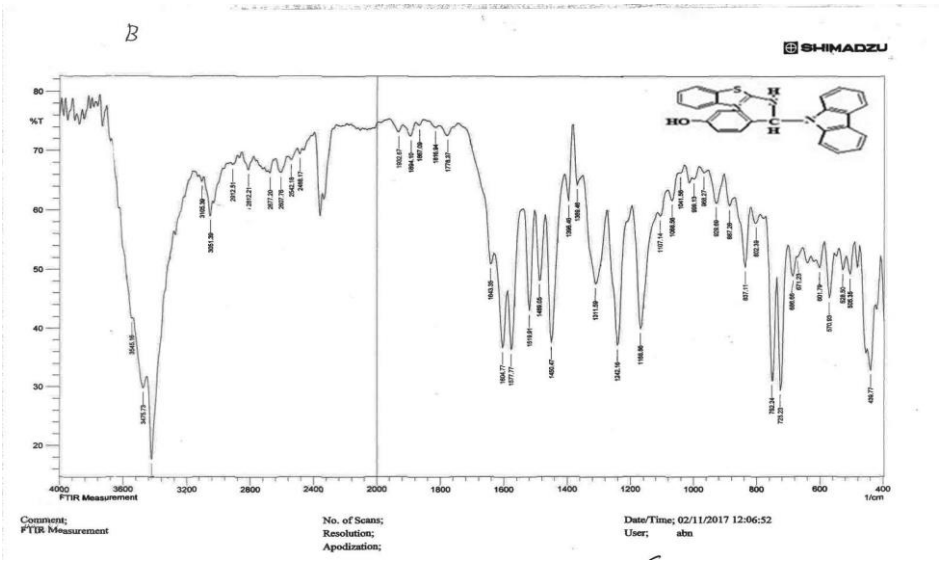

Figure 1. The FTIR results for prepaid material (B)

The result that presented in Fig. 2 which showed compound (E) FTIR spectra. The result approved showed absorption of υ (NH2) for amine at 3421, 3317 cm^{-1} and 3468 owing to υ (O-H). Additional packs occurred at 1636,3116,1462,3012, and (2931, 2831) cm^{-1}. That packs are due to present of U(C=N), U (N-H), U(C-N), U (C-H) aromatic, and U(C-H) aliphatic sequentially

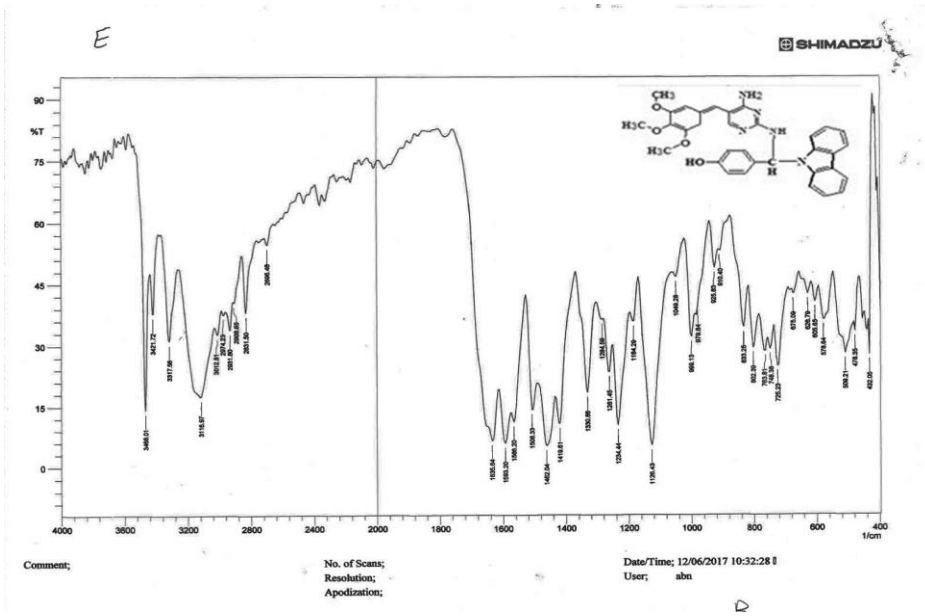

Figure 2. The FTIR results for prepaid material (E)

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1H-NMR spectra were used to verify the chemical shifts, multiplications, and pairing constants of the competently prepared derivatives.

Fig. 3 gives a presentation of the chemical (A[(1H-NMR))'s spectrum], The results showed signals at phenyl proton δ 6.91-8.21 ppm. 1H proton of NH-C=O at δ 9.79 ppm, 2H protons of (CH_2-N) were at around δ 3.53 ppm. 1 H proton of OH at δ 8.25 ppm, 1H proton of CH-N at δ 6.03 ppm, 3 H protons at CH_3- at δ 1.28 ppm, and 2H protons of CH_3-CH_2-N, δ 2.27-2.73 ppm, 3 H protons at CH_3-O, δ 3.84 ppm.

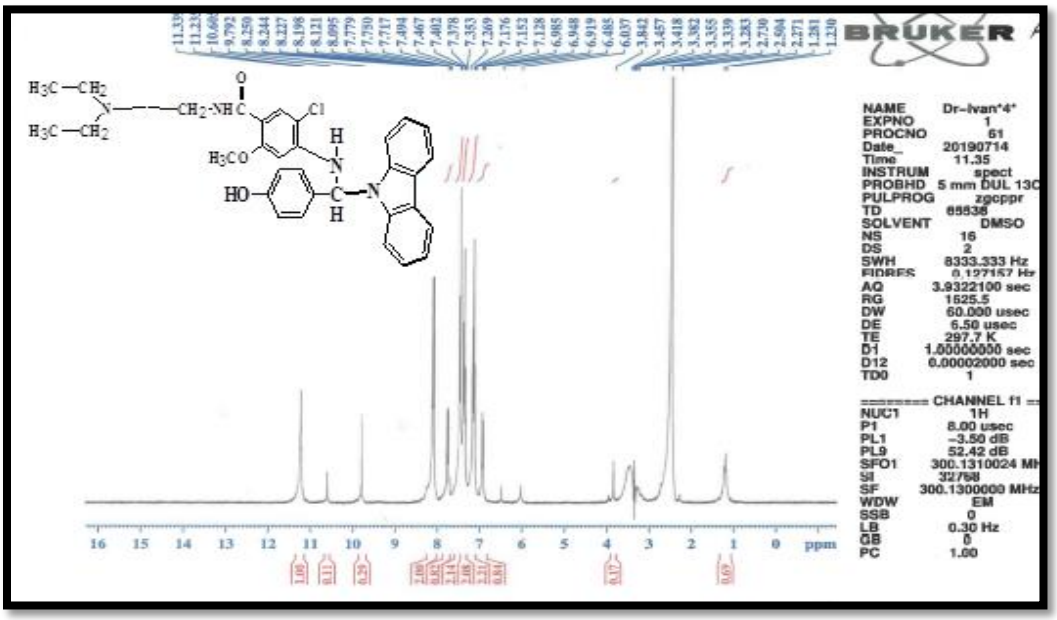

Figure 3. The results of (1H-NMR) spectral for synthesized materials (A)

So (1H-NMR) spectrum of compound (D) Fig. 4 exhibited signals in δ 6.95-8.12 ppm return to aromatic ring protons δ (6.55) ppm return to (-NH), appeared signals in δ (5.76)ppm belong to 4 H protons of (-CH_2-CH_2-). δ 2.72 ppm due to N-CH3 , δ 1.23 ppm due to (-CH_3), 8.36 due to OH phenolic group, and 3.50 due to 1 H protons for N-CHN . δ 3.54 due to (-CH_2-CH_2-).

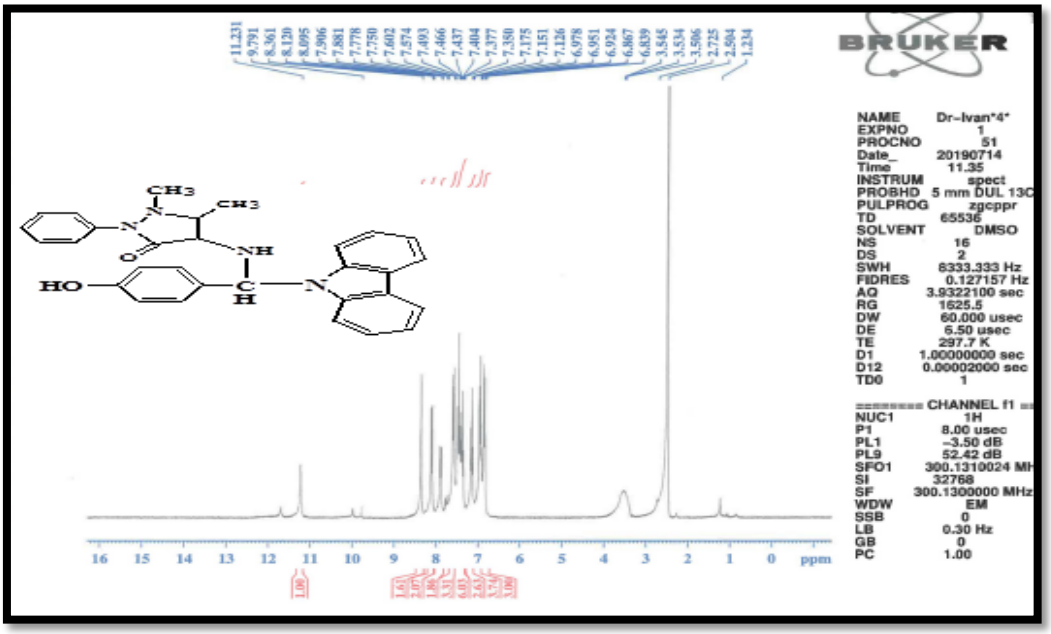

Figure 4. The results of (1H-NMR) spectral for synthesized materials (D)

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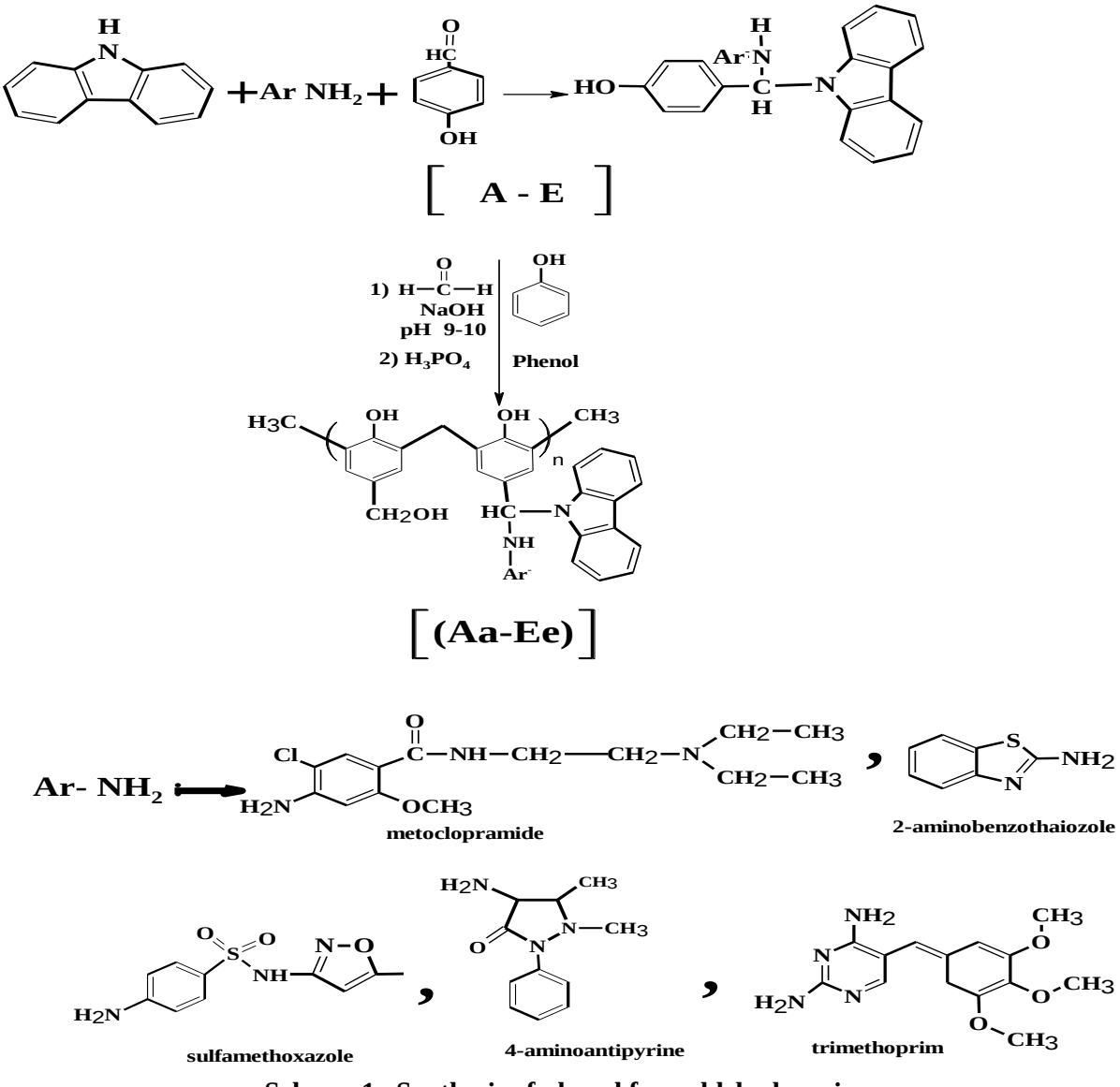
Section Two was done through applying polymerization predominantly batch polymerization using phenol, and formaldehyde with the previously made Mannich bases in section one, phenol formaldehyde (P.F.) resin (phenolic chelating polymers) was created, Scheme. 1.



Scheme 1. Synthesis of phenol formaldehyde resin

Several parameters were affects resin organization. The most common is a degree of condensation with affecting how the resin is organized as the aromatic phenol ring, involves the [ortho and para] positions that can condense with formaldehyde, but the para location is further likely to do so than the ortho position. In a typical aromatic ring, the presence of one para location and two ortho locations can result in the production of resin via the phenol process that used (phenol-formaldehyde). The reagents are able to produce additional formaldehyde resin with [ortho hydroxyl-methyl] groups particularly. Therefore, excess ortho on para substituted positions in formaldehyde or methylol towards phenol (ortho) positions lead to more responsive useful groups or more unreacted par appositions in the period of handling treatment, which may shorten the time of treatment and rise the degree of cross-linking for cured {phenol formaldehyde} resin.[1,3]

Physical characteristics and spectroscopic data are typically used to define novel phenol formaldehyde resin derivatives in Tables 2 and 3.

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Table 2. Physical properties of prepared Mannich bases (A-E)

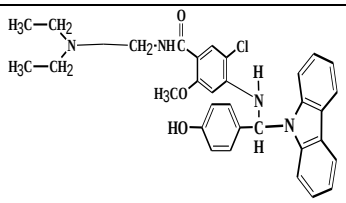
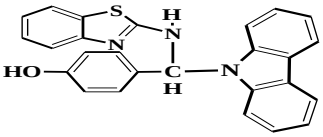
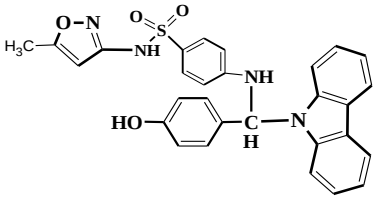
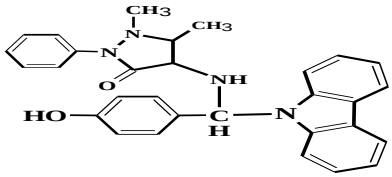
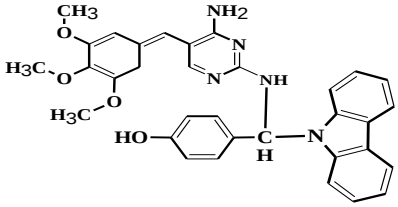
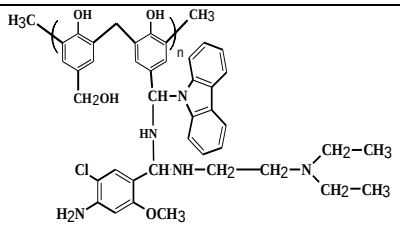
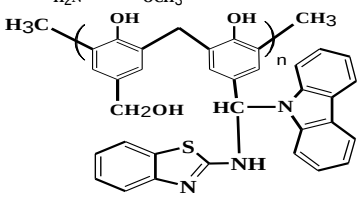
Comp. No.	Molecular Structure	Color	Yield %	FW	M. P. C°	Molecular weight
A		Brown	60	$C_{33}H_{35}ClN_4O_3$	147-149	313.82
B		Yellow	70	$C_{26}H_{19}N_3OS$	165-167	150.2
C		Light brown	76	$C_{10}H_{11}N_3O_3S$	148-150	253.27
D		white	65	$C_{30}H_{28}N_4O_2$	175-177	205.25
E		Light brown	40	$C_{33}H_{31}N_5O_4$	152-155	292.29

Table 3. Physical properties data of prepared polymers (Aa-Ee)

Polymer No.	Polymer Structure	Color	Yield %	Molecular weight for repeated unit	Softing Point C^0	Molecular formula for repeated unit
Aa		Dark gray	80	706.34	215-218	$C_{41}H_{44}ClN_5O_4$
Bb		Pale red	85	527.66	190-192	$C_{34}H_{25}N_3O_3S$

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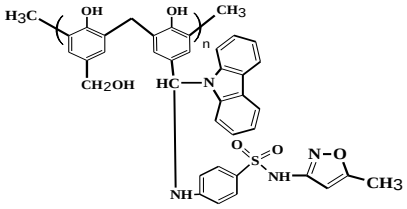
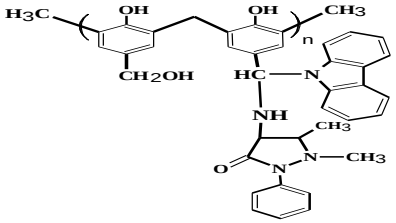
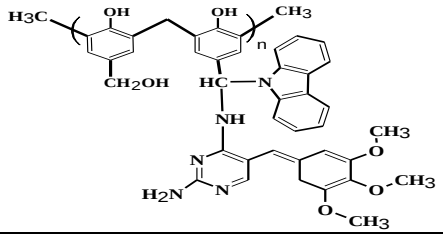
Cc		Red	80	658.74	175-179	$C_{37}H_{30}N_4O_6S$
Dd		Dark Brown	75	610.72	205-208	$C_{38}H_{34}N_4O_4$
Ee		Brown	70	695.29	185-188	$C_{41}H_{37}N_5O_6$

Compound (Bb) FT-IR spectra Bands were presented in 3045, (2985,2831),1600, 3211,3309, and 1450) cm^{-1} the results are shown in Fig. 5, the spectra bands were due to υ(C-H) arom, υ(C-H) aliph , υ(C=C , stretching band for υ(N-H) , υ(O-H , and υ(C-N) .

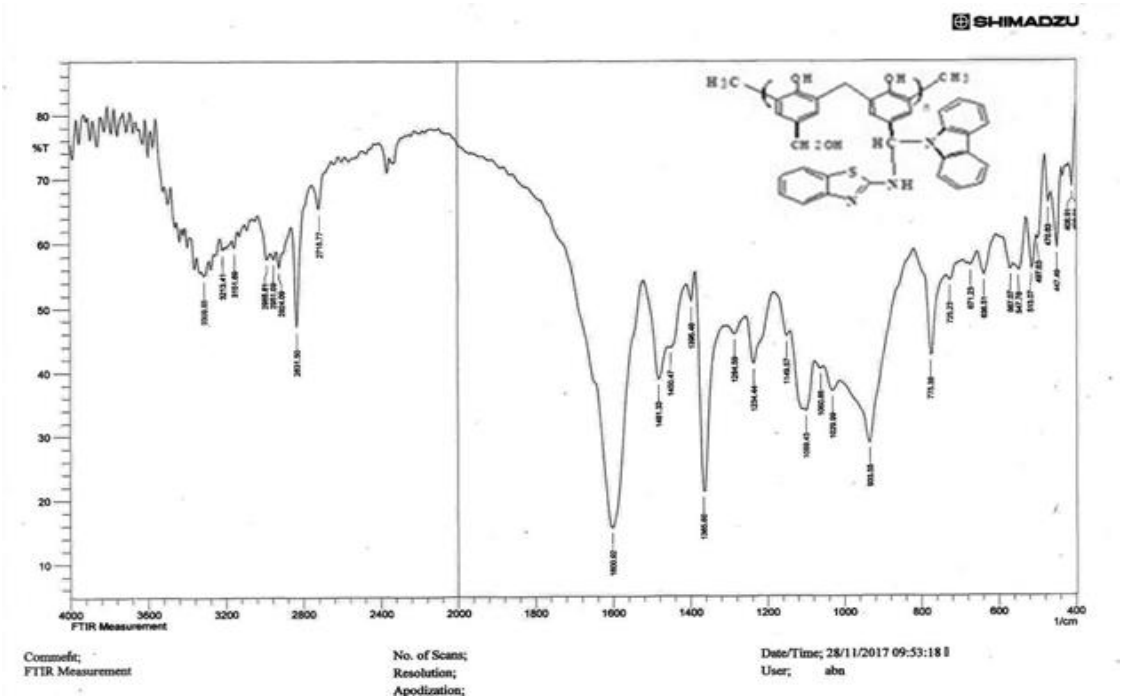


Figure 5. The FTIR results for prepaid materials (Bb).

Compounded similarly (Ee), Fig. 6 displays bands at 3039, (2924, 2838),1651, 3267, 3430 and 1477 cm^{-1} due to υ (C-H) arom., υ (C-H) aliph., υ (C= N), υ (N-H), υ (O-H) and υ (C-N) successively.

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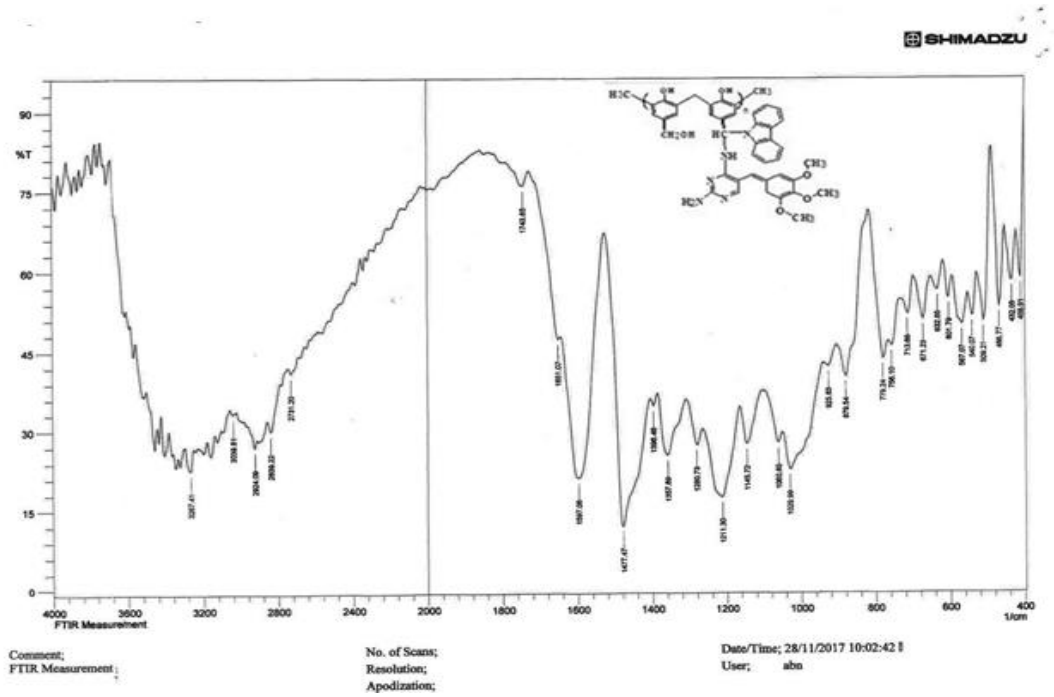

Figure 6. The FTIR results for prepaid material (Ee).

Fig. 7 offers an illustration of the compound (Aa),[1]H-NMR spectrum revealed signals at δ (5) ppm due to the υ (OH) phenolic group, δ (6.90-7.10) ppm due to aromatic-ring-protons, δ 8.51 ppm} due to the υ {-NH) , and δ (7.14-7.73) ppm due to the 4H protons for N-CH_2-CH_2N. Moreover, signals for the CH_2-OH) proton and (-CH2-) proton, respectively, occurred at (1.30) ppm and δ(4.50-4.78)ppm .

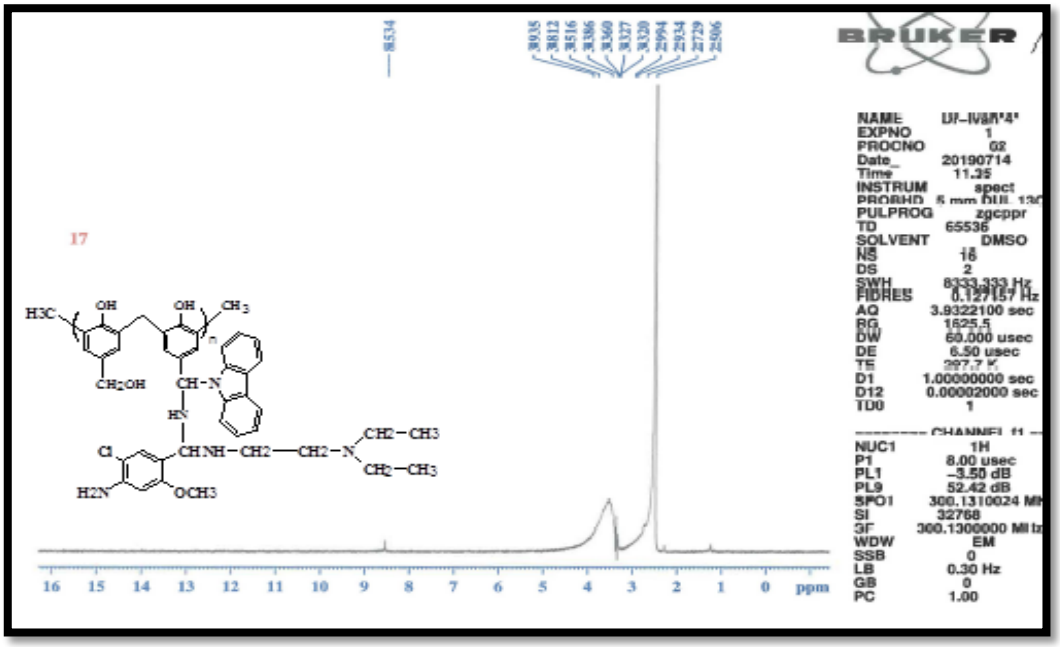

Figure 7. The results of (1H-NMR) spectral for prepared complex (Aa)

The results of compound (Dd) presented [1]H-NMR spectral data in Fig. 8, the results display signals in δ(6.97-7.72) ppm belonging to aromatic-ring-proton, δ(9.57) ppm, due to OH phenolic group, δ 6.58 ppm due to (-NH), δ(3.70) ppm because of (O-CH_3). Consequently, signals exposed at δ 2.18-2.41 ppm belonging to (-CH_2-) proton and δ(4.50-4.78) ppm belonging to (CH_2OH) proton.

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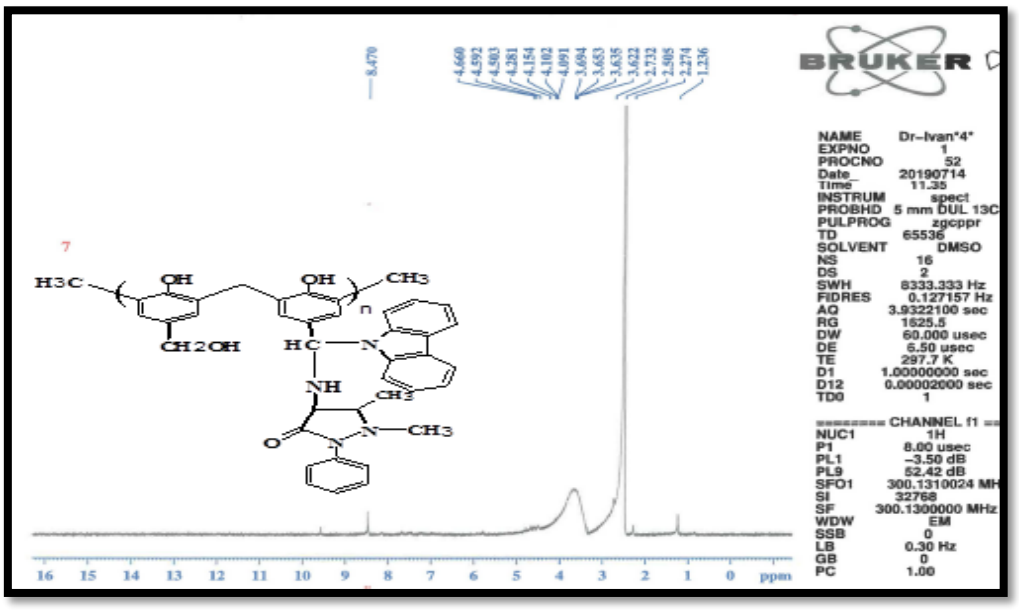

Figure 8. The results of (1H-NMR) spectral for prepared complex (Dd)

Table 4. (FT-IR) spectrum for all product compounds

Com. No.	υ C-H arom. cm^{-1}	υ C-H aliph. cm^{-1}	υ C=C cm^{-1}	υ N-H cm^{-1}	υ O-H cm^{-1}	Y C-N cm^{-1}	Others cm^{-1}
A.	3047	2877	1600	3170	3417	1450	C=O 1670
B.	3051	2812	1604	3105	3475	1489	(C=N) ring 1643
C.	3051	2885-2981	1620	3143	3417	1450	(C=N) ring 1652 (S=O) 1285
D.	3051	2893	1604	3255	3417	1450	(C=O) Amid 1670
E.	3012	2831	1600	3116	3468	1462	(C=N) 1636
Aa.	2951	2831	1597	3151	3300	1477	(C=O) 1647
Bb.	2985	2831	1600	3212	3309	1450	(C=N) 1481
Cc	2951	2831	1597	3151	3305	1481	(C=N) ring 1643 (S=O) 1222
Dd.	2978	2831	1593	3217	3448	1477	(C=O) amid 1750
Ee.	3039	2924	1597	3267	3430	1477	(C=N) 1651

Section Three

Anti-bacterial activity is presented in part three. The activity and application of some generated materials were investigated using two types of bacteria gram positive and gram negative *Staphylococcus aureus, Bacillus subtillus, Escherichia coli, Klebsiella ssp., Acintobacter baumannii* as well as *Rhizosporium*. The results are presented in Table 5. Biological

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activity was used to regulate the biological efficacy for potential use as pharmaceuticals or the rapeutic[11, 12]. In DMSO, which also acted as a control, all Mannich bases were dissolved. The findings demonstrated that created compounds have superior action against [gram positive and gram negative] bacteria than all other compounds developed for compound (4)[13]. Table 5 displays the potency of produced compounds counter to particular species in the absence of other species[14]. Various inhibitory zones of varying sizes were seen for the investigated bacterial strains Fig. 9-18. Compound (4) displayed good and highest efficacy against all species of bacteria as well as Rhizosbarium thanks to the action of resin components. The inhibition zones that were created by each medicine under investigation are presented in Table .5.

Table 5. The Results of Biological activity for some synthesized materials.

Compound	The inhibition zone against some bacteria in mm at 10 mg/ml				
	Gram positive		Gram Negative		*Rizosporium*
	S. aureus	***B. subtilis***	***E. coli***	***Klebsiella* ssp.**	
A	-Ve	16	-Ve	17	-Ve
C	30	17	40	-Ve	13
D	-Ve	14	-Ve	16	11
E	12	20	50	-Ve	12
Aa	11	17	33	16	13
Cc	19	16	26	17	12
Dd	12	15	21	18	10
Ee	13	15	18	17	12
Carbazole	15	14	18	16	13

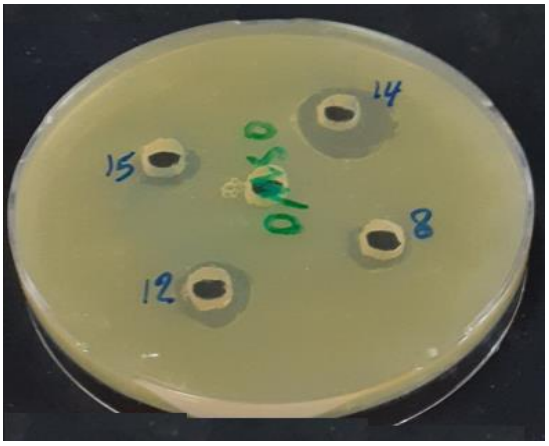

Figure 9. Effect of compound (8, 12, 14, 15) in (staphylococcus aureus)

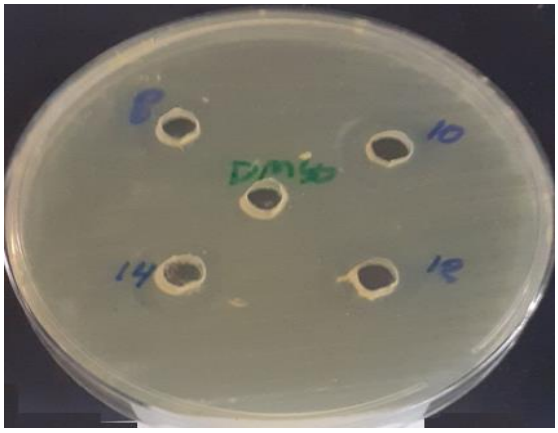

Figure 11. Effect of compound (9, 11, 13, 18) in (staphylococcus aureus)

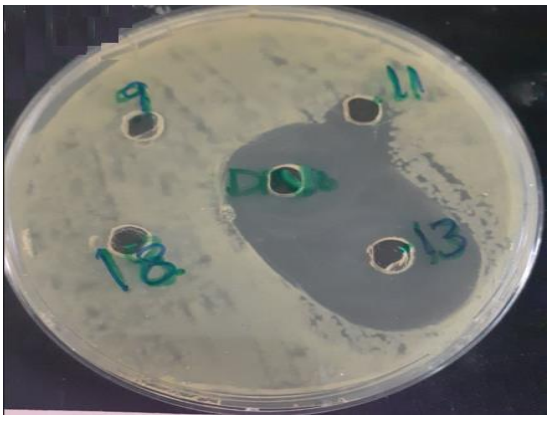

Figure 10. Effect of compound (9, 11, 13, 18) in (staphylococcus aureus)

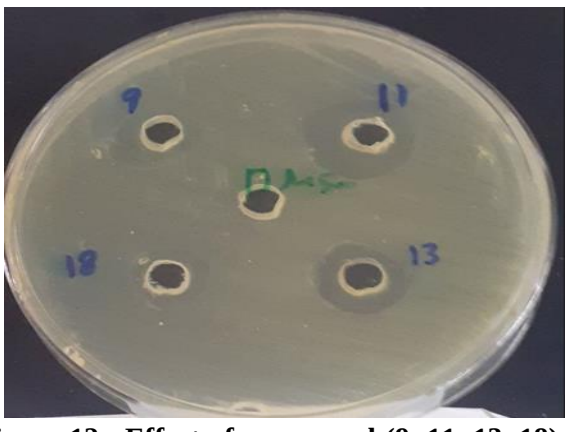

Figure 12. Effect of compound (9, 11, 13, 18) in (bacillus subtillus)

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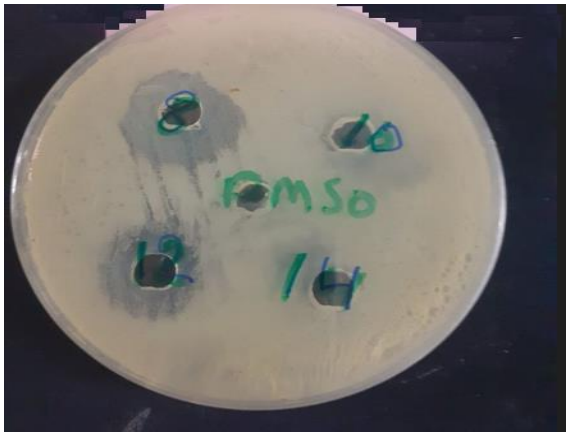

Figure 13. Effect of compound (8, 10, 12, 14) in (eschericha. coli.)

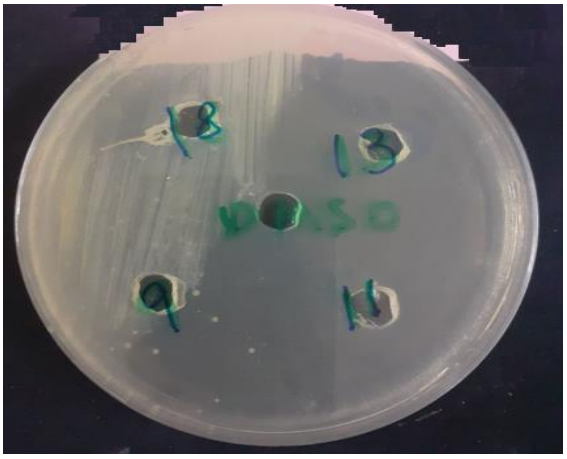

Figure14. Effect of compound (9, 11, 13, 18) in (eschericha. coli.)

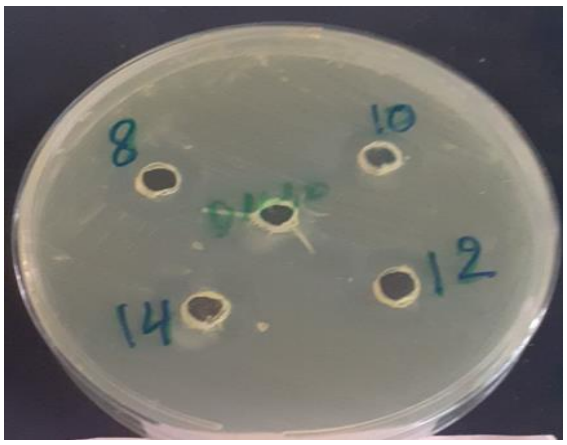

Figure 15. Effect of compound (8, 10, 12, 14) in (klebsiella spp.)

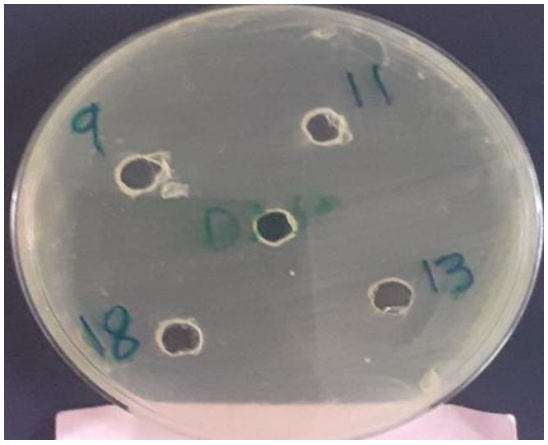

Figure 16. Effect of compound (9, 11, 13, 18) in (klebsiella spp.)

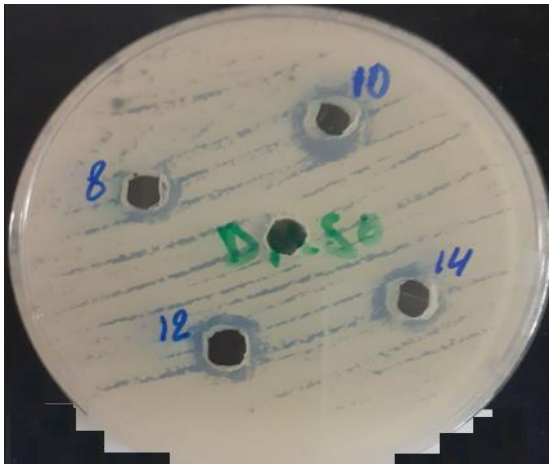

Figure 17. Effect of compound (8, 10, 12, 14) in (rhizo.)

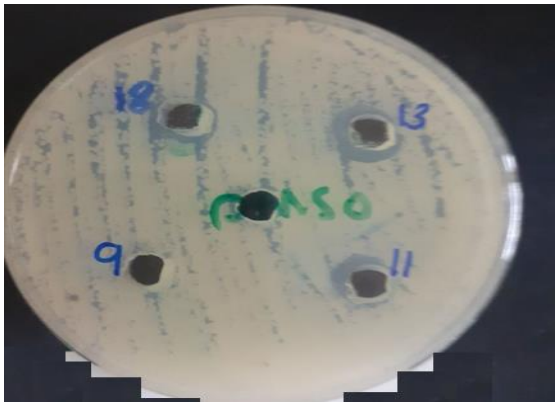

Figure 18. Effect of compound (9, 11, 13, 18) in (rhizo.)

Conclusion

A new Mannich bases were created through the reaction of caebazole, parahydroxybenzaldehyde, and various primary amines (1–5). The project was divided into two phases. The first portion involves creating a monomer of Mannich bases through condensation reaction. The reaction occurs between aromatic aldehyde (P-hydroxy benzaldehyde) and primary amines like (1-5) with minor amines such as

Carbazol(A-E). By condensing Mannich bases made in part one with formaldehyde and phenol, the second stage included making phenol formaldehyde resin with Mannich bases (Aa-Ee) . The synthesized resins (Phenol-formaldehyde) have been characterized.

Ultimately the examinations of FTIR, H-NMR and 13C.NMR provided proof that the chemicals were prepared and also that to determine the degree of biological activity associated with some of the active groups and their antifungal properties were tested.

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Authors' Declaration

- Conflicts of Interest: None.
- I hereby confirm that all the Figures and Tables in the manuscript are mine. Furthermore, any Figures and images, that are not mine, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- Authors sign on ethical consideration's approval.
- Ethical Clearance: The project was approved by the local ethical committee at University of Baghdad.

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تحضير وتشخيص بعض الراتنجات الدوائية بأستخدام قواعد مانخ ودراسة الفعالية البيولوجية لها

تماضر علي محمود

قسم الكيمياء، كلية العلوم للبنات، جامعة بغداد، بغداد، العراق.

الخلاصة

تم تحضير راتنج الفينول فورمالديهايد باستخدام قواعد مانخ للمجموعات الوظيفية والتي تعتبر مبتكرة في هذه الدراسة. يتضمن البحث مرحلتين. يتضمن الجزء الأول إنشاء مونومر لقواعد مانخ من خلال تفاعل التكثيف. يحدث التفاعل بين الألدهايد الاروماتي (باراهيدروكسي بنزلديهايد) والأمينات الأولية مثل (ميتوكلوبراميد ، تريميثوبريم ، 2-أمينوبنزوثيازول ، سلفاميثوكسازول ، و 4-أمينو أنتيبيرين) مع أمينات ثانوية مثل كاربازول. من خلال تكثيف قواعد مانخ المصنوعة في الجزء الاول بالفورمالديهايد والفينول , تضمنت المرحلة الثانية تحضيراتنج الفينول فورمالدهيد مع قواعد مانخ تم تمييز الراتنجات المركبة (الفينول فورمالديهايد). تم التشخيص بواسطة (H-NMR1 ، و FT-IR)بحيث يتم تحديد درجة الفعالية البيولوجية المرتبط ببعض المجاميع النشطة.

الكلمات المفتاحية: النشاط البيولوجي، كاربازول ، قواعد مانخ ، الراتنجات الصيدلانية ، فينول فورمالدهيد.