SYNTHESIS, CHARACTERIZATION AND CYCLAZATION OF PYRANUSING Ag2O NANOPARTICLE FROM NATURAL SOURCE "GINGER"Ruaa M. D.*S. A. AlsahibI. F. AscarLecturerAssist. Prof.Lecturer

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ABSTRACT

This study was aimed to synthesis Ag₂O nanoparticles (N.P.s) utilizing the Ginger plant (*Zingiber officinale*) extract. As catalysts for pyran derivatives formation employing a threecomponent coupling reaction among aromatic and aliphatic aldehydes, malononitrile, and dimedone. The nanoparticles exhibit robust catalytic activity with high productivity. Results revealed that Ag₂O NPs improved various vital features, like higher yields, reaction time, simple chemical separation, catalytic economic efficiency, and quick process. This study aimed to cyclize heterocyclic compounds to provide new hetero compounds applying nanooxides obtained from natural (unmanufactured) sources and used in several medical, pharmaceutical, and industrial implementations. The outcomes nanoparticles were examined by Fourier Transform Infrared spectroscopy (FTIR) and transmission electron microscopy (T.E.M.); the chemical characteristics of the outcome compounds were tested by spectroscopy techniques (FTIR spectroscopy and 1H-NMR).

Key words: nano oxides, Schiff bases, heterocyclic compounds, FTIR, HNMR.

مجلة العلوم الزراعية العراقية -2021 :55 (5):1184-1171 فيدان وآخرون تحضير وتشخيص لمشتقات البايرين باستعمال اوكسيد الفضة النانوي والمحضر من مصدر طبيعي (نبات الزنجبيل) رؤى محمد ضيدان سناء عبد الصاحب عبد الكريم اسراء فاضل عسكر مدرس أستاذ مساعد مساعد مدرس قسم الكيمياء - كلية العلوم للبنات - جامعة بغداد

المستخلص

تهدف الدراسة الى تحضير جسيمات الدقيقة لمادة اوكسيد الفضة Ag₂O باستخدام مستخلص نبات الزنجبيل كعامل مساعد لتحضير مشتقات البايرين المكونة باستخدام تفاعل الاقتران الثلاثي من بين مجموعة من الالديهايدات الاروماتية والالفاتية كالمالوتونتريل والدايمودون. وقد اظهرت الجسيمات الدقيقة فعالية قوية كعامل مساعد مع نسبة عالية من الانتاج. كما اظهرت الجسيمات الدقيقة بان دقائق اوكسيد الفضة قد حسنت كمية المنتوج ايجابيا ووقت التفاعل وبساطة الفصل الكيميائي والكفاءة الاقتصادية للعامل المساعد. ان اللهدف من دراستنا هو تحضير المركبات الحلقية غير المتجانسة باستخدام جسيمات الدقائق التي تم الحصول عليها من مواد طبيعية غير مصنعة في مجالات متعددة كالمجال الطبي والصيدلاني والصناعي. وقد تم تشخيص جسيمات الدقائق بواسطة الاشعة تحت الحمراء والانتقال الالكتروني كذلك تم تشخيص المركبات المحضرة بواسطة التحليل الطيفي بالأشعة تحت الحمراء والانتقال الالكتروني كذلك تم تشخيص المركبات المحضرة بواسطة

الكلمات المفتاحية: الجسيمات النانوية، قواعد شف، المركبات الحلقية الغير متجانسة، التحليل الطيفي بالأشعة تحت الحمراء، التحليل الطيفي بالرنين النووي المغناطيسي لذرة الهيدروجين.

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INTRODUCTION

Nanotechnology is of growing interest in several science fields due to its miniaturization capacity and the essential characteristics related to small particle size (1). Nano-metal oxide usages in synthetic organic chemistry have been confirmed attractive due to their excellent chemical and physical characteristics (8). High oxidizing power, recall capability, high behavior, and long-term stability are these nano metal oxides (18). Noncrystalline metal oxides such as nanosilver oxide (Ag₂O) nanoparticles is a famous material having extensive usage regarding sensors (25), oxidation catalysis (13, 32),all-optical switching devices, optical data storage systems (20),diagnostic biological probes (33), photovoltaic cells (16) and fuel cells (28). The antifungal, antibacterial, and antiviral functions of spice plants are now underlined in various studies (26,6). (Solanum tricobatum, Ocimum tenuiflorum, Centella asiatica, Syzygium cumini, and Citrus sinensis were examined to prepare silver nanoparticles from silver nitrate solution. S. tricobatum, O. tenuiflorum extracts were showed the highest antimicrobial activity of silver nanoparticles against S. aureus and E. coli. (21). Syzygium aromaticum (Eugenia caryophyllus) was used to synthesized Copper Oxide (CuO) as a nanoparticle (NPs) of 17-22 nm size. The CuO NPs were utilized as a nano-catalyst for the synthesis polymerization and of 1Hpyrazolo[1,2-b]phthalazine-5,10-dione derivatives three-component coupling reaction between malononitrile and aldehydes, 5,5diethyl-2,3-dihydrophthalazine-1,4(5H,8H)dione. Results showed that CuO NPs enhanced several significant points positively, such as reaction time, catalytic economic efficiency, simple chemical separation, quick process, higher yields, and no harmful by-products (4). The Pyran family has been confirmed to be the primary organic compound that shows infectious, antitumor, antibacterial, antiallergic and immune-modulating activity. $(22)^{-}$

Zingiberaceae plants were revealed for their curative applications (7). Zingiber officinale is the most important medicinal plant, and it has possessed several medicinal values. Zingiber officinale, mostly knowns as Ginger. This crop's rhizome is mostly utilized in the formulation of folk medicine and as a nutraceutical well. In Chinese and Ayurvedic conventional habits of medicines, Z. officinale has been applied for curing arthritis, microbial infections, fever, rheumatism (10). Imine (Schiff base) is essential group in demonstrating transformation in biological activities (35). Schiff base compounds' ability to construct heterocyclic compounds, like 1,3thiazolidin-4-one using mercaptoacetic acid (24). Thiazolidin-4-one cyclic systems are of major interest as it is a substantial structure in several synthetic pharmaceuticals that show a broad spectrum of biological actions (12), such anti-HIV (27), antifungal as (23),(antheltminitic, antiviral, antibacterial and antitubercular, antihistaminic (H1-antagonist), (31). Therefore, using an inexpensive and mild catalyst utilizing modern catalytic strategies, such as nanoparticles to construct like these bioactive molecules. Due to its unique characteristic of interactive surfaces. reusability, and selective reactivity, nanocatalysis has lately drawn considerable interest study was directed toward This (30). synthesizing heterocyclic compounds from Schiff base by using nano oxides obtained from natural sources (plants) and utilized in many pharmaceuticals and medical purposes.

MATERIALS AND METHODS

Chemical: Industrial manufacturers are the source of reagents that were collected and used without purification. Using open capillary tubes, melting points were calculated using the system FP800 Mettler central Thermo processor provided with the FP81 MBC cell devices (Stuart Research, Redhill, U.K.) were not corrected. Infrared spectra (Using FTIR Shimadzu (Japan)) were calculated as KBr disks in the range of (400 -4000 cm⁻¹) at Ibn Sina Company (Iraq-Baghdad) and FTIR Smoothing in BPc-Analysis Center. The δ (part per million-ppm) is the unit for the chemical change relative to the residual solvent peak; also, coupling constants (J) in Hertz (Hz) were recorded. ¹H-NMR data were calculated in Tehran University of Iran, Spectra utilizing DMSO-d6 as a solvent on a 400 MHz Bruker appliance.

Synthesis of nanoparticles of Ag₂O: ⁽¹⁷⁾

Preparation of plant extract from the root of Zingiber officinale: The extract was prepared by weighting (0.4g) of the seeds (Zingiber officinale), washing with distilled water to clear impurities. Then, 200 mL of distilled water was added to the seeds, heated with stirring up to 70 °C, and centrifuged for 10 minutes. The supernatant was then filtered twice and stored at 4.0 °C for further use, Figure (1, 2).



Figure 1. The color change of the Ag₂O NPs solution



Figure 2. Formation of Ag₂O NPs due to change in the color to dark

Nanoparticles catalyzed synthesis of pyran derivatives compounds [C1], [C2], [C3]^(36,19)

different А mixture of aldehyde {propionaldehyde, 4-(dimethylamino) 4-nitrobenzaldehyde} benzaldehyde, (0.01)dimedone (0.130gm, mol). 0.01 mol). malononitrile (0.066gm, 0.01 mol), and Nano Ag₂O catalyst (0.003 gm), in ethanol (10 mL) was refluxed for 1h. T.L.C followed the progress of the reaction. After the completion of the reaction, the mixture was filtered to remove the catalyst, and the crude product was

recrystallized from ethanol to obtain the pure compound [C₁], [C₂], and [C₃]. Two drops of H_2SO_4/H^+ were added to the end with hydrolysis, (Table 1).

Preparation of Schiff Bases derivatives 7,7dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-

chromene-3-carboxylic acid derivatives: [C4], [C5], and [C6] ^(3, 2, 5)

A mixture of compounds C1, C2, and C3 (0.001mmol) was added to different aldehydes (p-chlorobenzaldehyde, 4-dimethylbenzaldehyde, and o-hydroxybenzaldehyde) in absolute ethanol, and then a few drops of glacial acetic acid were added. The mixture was refluxed for 3hrs and then cooled. The precipitate was collected then recrystallized from ethanol. T.L.C. monitored the completion of the reaction to give the product [C4], [C5], and [C6], Table (1).

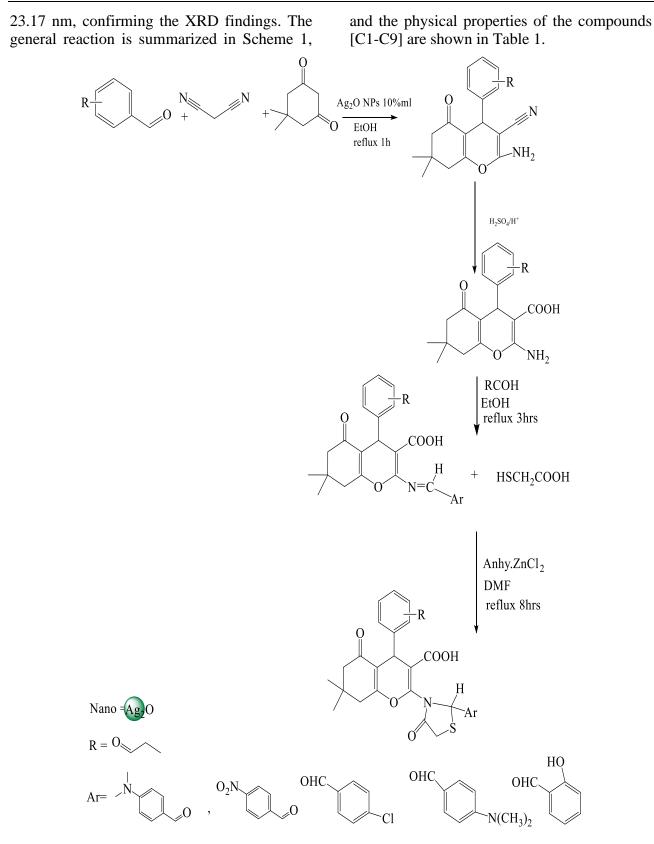
The synthesis of 1,3-thiazolidine- 4-ones derivatives [C7, C8, and C9]⁽¹¹⁾.

A mixture of the compounds C4, C5, C6 (0.01mol) and (0.012 moles) of mercapto acetic acid in (25 mL) of DMF containing a little amount of anhydrous ZnCl₂ was refluxed for 8 hrs. The mixture was cooled and then poured into the icy water. The mixture was filtered and washed many times with water and then crystallized from DMF to produce [C7], [C8], and [C9] Scheme-1, (Table 1).

RESULTS AND DISCUSSION ⁽²⁹⁻³⁴⁾

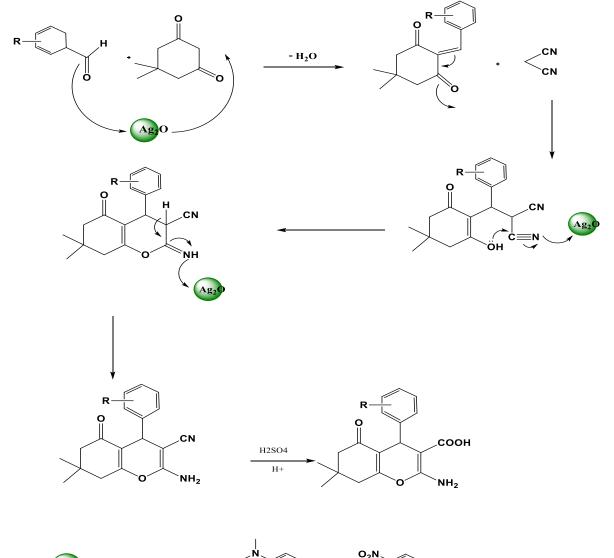
Characterization of nanoparticles of copper oxide:

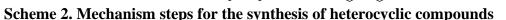
FTIR spectrum (5) of Ag₂O nanoparticles is shown in Figure3. Feature peaks of Ag₂O range between 443 cm⁻¹ and 1000 cm⁻¹. Peaks between 443 cm⁻¹ and 961cm⁻¹ can be assigned to stretching along with the Ag₂O trend. A small sharp peak around 486.06 cm⁻¹ is due to Ag₂O stretching along with the trend. The peak around 2357.09 cm⁻¹ can be attributed to CO₂ stretching, which could have been entrapped in the sample holder during packing. The overall particle size and the form of the particle Ag₂O N.P.s have been identified, and the analyzed usage (T.E.M.) indicated the distribution of nanoparticle form in Figures 4 and 5. The mean diameter of Ag₂O N.P.s was determined from the calculation of over 100 particles in the random field of view of the T.E.M. The average T.E.M. size of the Ag₂O N.P.s was about



Scheme 1. Summarized the reaction.

Compound [C1] was synthesized by interacting malononitrile with propionaldehyde and dimidone under (10 mol) nano-Ag₂O. FT-IR spectrum (29) was used to diagnose the structure of compound [C1], which revealed a stretched vibration of (NH2) group (3387- 3375) cm⁻¹ related to v (NH₂) asymmetric and symmetric, v(C-H) aliphatic at (2981- 2935), v(C-H) aromatic at (3055), v(C=C) at (1618), v(C=N) at (2225). Moreover, many other bands are explained in Table 2. Compound [C2] was synthesized by interacting malononitrile with 4nitrobenzaldehyde and dimidone under (10 mol) nano-Ag₂O. FT-IR spectrum (29) was used to diagnose the structure of compound [C2], which revealed a stretched vibration of (NH₂) group (3475-3437) cm⁻¹ related to v (NH₂) asymmetric and symmetric, v(C-H) aliphatic at (2850), v(C-H) aromatic at (3024.38), v(C=C) at (1670) v(C=N) at (2235). Moreover, many other bands are explained in Table 2. Compound [C3] was synthesized by interacting 4-(dimethylamino) benzaldehyde and dimidone under (10 mol) of nano-Ag₂O. The structure of compound [C3] was diagnosed by FT-IR spectrum (29) showed the appearance of stretching vibration of (NH₂) group (3205-3174) cm⁻¹ belongs to v (NH2) asymmetric and symmetric, v(C-H) aromatic at (3006), v(C-H) aliphatic at (2974-2939), v(C=C) at (1610), v(C=N) at (2228), and several other bands are described in Table 2.





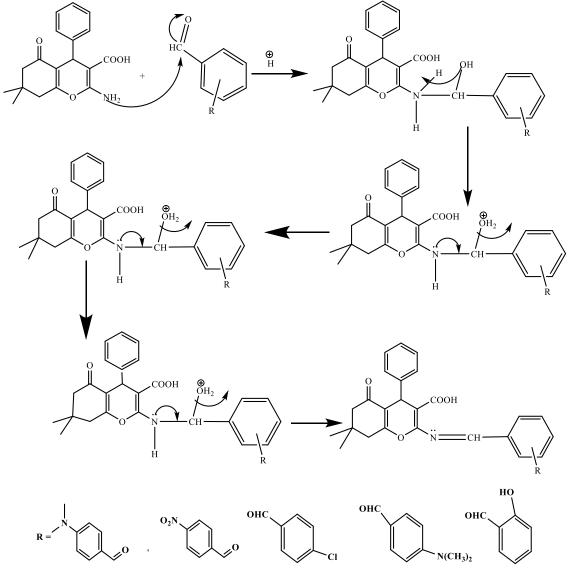
Compound [C4] was synthesized by interacting one mmole of compound [C1] with one mmole of 4-dimethylamino benzaldehyde in the solvent of absolute EtoH, where the mixture stirred for 3.0 hrs. FT-IR spectrum (29) was used to diagnose the structure of the compound [C4]. These spectra revealed the disappearance of bands of the NH₂ group and

Nano=

the apparition of bands (C=N) at (1622) cm⁻¹, υ (O.H.) acid at (3483) cm⁻¹, and the disappearance of bands to (C=N) at (2225) cm⁻¹. The apparition of bands v(C-H) aliphatic at (2976-2821) cm⁻¹, v(C-H) aromatic at (3062.96) cm⁻¹, and v(C=O) acid at (1730) cm⁻¹, Table 2, Figure 6. Compound [C5] was synthesized by interacting one mmole of

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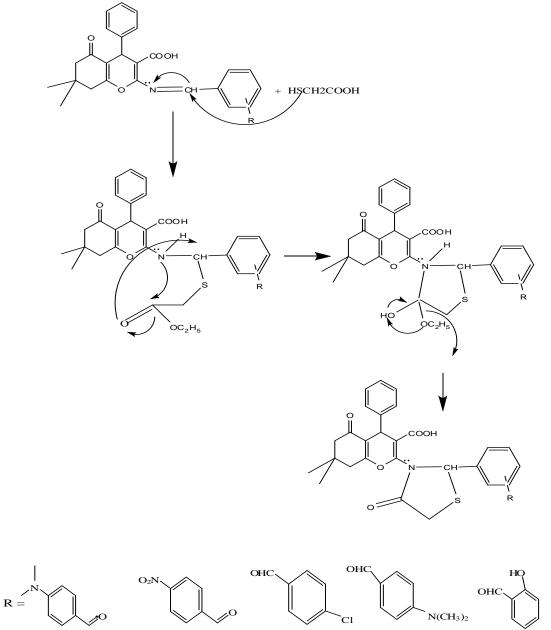
compound [C2] with one mmole of pchlorobenzaldehyde in absolute EtoH solvent; the mixture stirred for 3.0 hrs. FT-IR spectrum (29) was used to diagnose the structure of the compound [C5]. These spectra revealed the disappearance of bands of the NH₂ group and the apparition of bands (C=N) at (1662) cm⁻¹, υ (O.H.) acid at (3437) cm⁻¹, and the disappearance of bands to (C=N) at (2235) cm⁻¹. The apparition of bands v(C-H) aliphatic at (2908-2819) cm⁻¹, v(C-H) aromatic at (3035) cm⁻¹, and v(C=O) acid at (1730) cm⁻¹,(Table 2). Compound [C6] was synthesized by interacting one mmole of compound [C3] with one mmole of 2-hydroxy benzaldehyde in absolute EtoH solvent; the mixture stirred for 3.0 hrs. FT-IR spectrum (29) was used to diagnose the structure of the compound [C6]. These spectra revealed the disappearance of bands of NH₂ group and the apparition of bands (C=N) at (1651) cm⁻¹, v (O.H.) acid at (3089) cm⁻¹, and the disappearance of bands to (C=N) at (2228) cm⁻¹. The apparition of bands v(C-H) aliphatic at (2974-2827) cm⁻¹, v(C-H) aromatic at (3035) cm⁻¹, and v(C=O) acid at (17015) cm⁻¹, Table 2, (9).



Scheme 3. Mechanism of Schiff Base by Amino group

Compound [C7] A thiazolidine compound was synthesized due to the compound's reaction [C4] with Mercaptoacetic acid in DMF solvent; the mixture refluxed for 8 hrs. Compound [C7] revealed an absorption band v(C-S-C) at v (1389-1396), v (OH) acid at (3460) cm⁻¹, v(C-H) aromatic at (3078), (C=O) acid at (1720.50), v(C-H) aliphatic at v (2897-2819); other absorptions compounds are detailed in Table 2, Figure 7. Compound [C8] A thiazolidine compound was synthesized due to the compound's reaction [C5] with Mercaptoacetic acid in DMF solvent; the mixture refluxed for 8 hrs. Compound [C8] revealed an absorption band v(C-S-C) at v (1373-1388), v (OH) acid at (3471) cm⁻¹, v(C-H) aromatic at (3045), (C=O) acid at (1730), v(C-H) aliphatic at v (2866-2927); other absorptions compounds are detailed in Table 2, Figure 8. Compound [C9] A thiazolidine compound was synthesized due to the compound's reaction [C6] with Mercaptoacetic

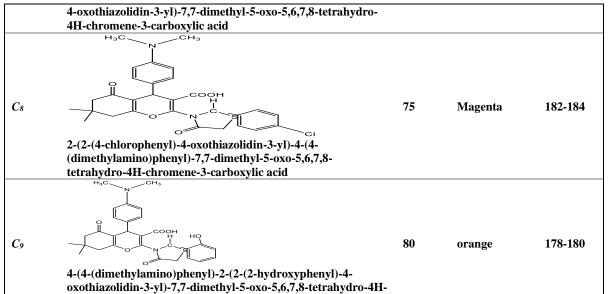
acid in DMF solvent; the mixture refluxed for 8.0 hrs. Compound [C9] revealed an absorption band v(C-S-C) at v (1342-1369), v (OH) acid at (3471) cm⁻¹, v(C-H) aromatic at (3086), (C=O) acid at (1735), v(C-H) aliphatic at v (2862-2924); other absorptions compounds are detailed in Table 2.



Scheme 4. Mechanism of a thiazolidine ring

Physical and spectroscopic data Table 1. Physical properties of the compounds prepared [C1-C9].

Table 1. Physical properties of the compounds prepared [C1-C9].								
Comp. No.	Nomenclatur & Structure formula	Yield%	Color	M.P.C				
<i>C</i> ₁	CH ₂ CH ₃ NH ₂ 2-amino-4-ethyl-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H- chromene-3-carbonitrile	75	yellow	190-192				
C ₂	2-amino-7,7-dimethyl-4-(4-nitrophenyl)-5-oxo-5,6,7,8- tetrahydro-4H-chromene-3-carbonitrile	80	Light yellow	214–218				
C3	2-amino-4-(4-(dimethylamino)phenyl)-7,7-dimethyl-5-oxo- 5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile	85	green	212-214				
C4	(E)-2-(4-(dimethylamino)benzylideneamino)-7,7-dimethyl-5- oxo-4-phenyloctahydro-2H-chromene-3-carboxylic acid	87	Dark red	184-186				
Cs	(E)-2-(4-chlorobenzylideneamino)-7,7-dimethyl-5-oxo-4- phenyloctahydro-2H-chromene-3-carboxylic acid	90	Magenta	150-152				
C6	(E)-2-(2-hydroxybenzylideneamino)-7,7-dimethyl-5-oxo-4- phenyloctahydro-2H-chromene-3-carboxylic acid	85	Violet	118-120				
<i>C</i> ₇	4-(4-(dimethylamino)phenyl)-2-(2-(4-(dimethylamino)phenyl)-2-(2-(4-(dimethylamino)phenyl)-	63	Magenta	190-192				



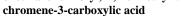
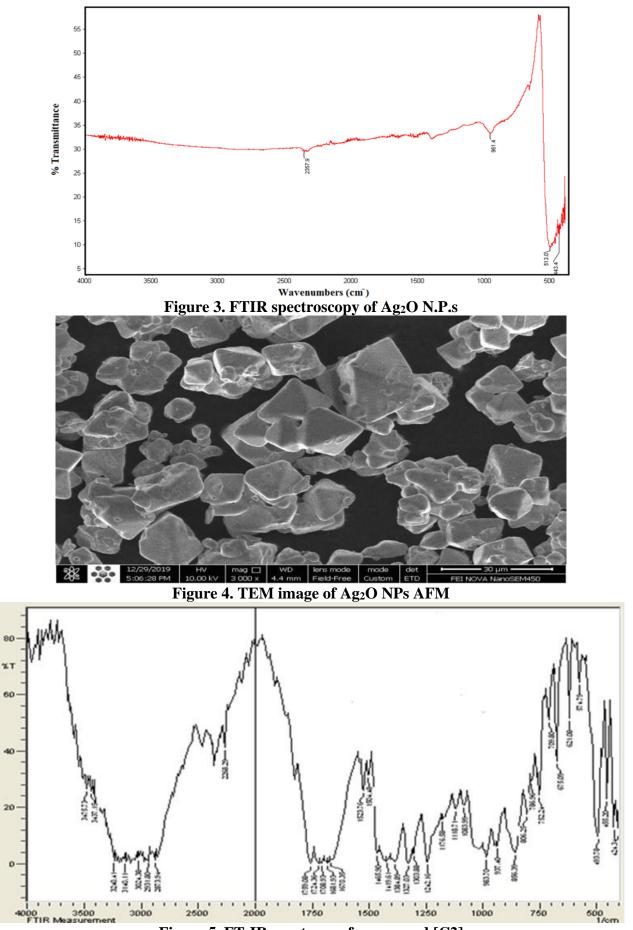
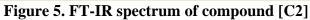


 Table 2. FT-IR spectrum of compounds [C1-C9]

comp	v (O.H.)	v (NH2)	v(C=N)	v(C-N)	v(C-H)	v(С-H)	v(C≡N)	v(C=O)	v(C=C)	v(C=O	Other
					aromatic	aliphatic		carbonyl)acid	bands
C1	3433	3387	/	/	3055	2981	2225	1700	1618	/	/
		3375				2935					
C2	3437	3475	/	/	3024	2939	2235	1759	1670	/	/
		3437				2850					
C3	3089	3205	/	/	3006	2974	2228	1715	1610	/	/
		3174				2939					
C4	3433	/	1622	1230	3084	2976,	/	1697	1602	1730	/
						2821					
C5	3437	/	1662	/	3035	2908,	/	1700	1602	1730	v(C-Cl)
						2819					817
C6	3089	/	1651	/	3035	2974,	/	1697	1600	1715	/
	3070 tau,					2827					
	ald										
Q7	3460	/	1	1242	3078	2897,	/	1689	1608	1720	v(C-S-C)
C						2819					1389-1396
C8	3471	1	/	1199	3109	2978,	/	1708	1604	1735	v(C-Cl)
00	0.1.1	,	,		0109	2885	,	1,00	1001	1,00	817.82
											v(C-S-C)
											1346-1450
C9	v(OH)	/	/	1185	3086	2924,	/	1690	1610	1735	v(C-S-C)
	3471					2862					1342-1369
	3429tau,										
	ald										

Table 3. 1H-NMR spectrum of compounds [C1-C9]. (34)						
Compounds	¹ H-NMR parameters δ (ppm)					
C_{I}	2.5(s,H,2CH ₃), 2.9(s,H,CH ₃), 3.33(s,H,2CH ₂), 2.63(s,H,CH ₂), 4.30(s,H,NH ₂).					
C_2	2.3(s,H,2CH ₃), 3.11(s,H,CH ₂), 3.7(s,H,CH ₂), 3.95(s,H,CH), 4.35(s,H,NH ₂), 7.07-7.8(m,H,Ar-H)					
C_3	2.63(s,H,2CH ₃), 3.00(s,H,CH ₂), 3.49(s,H,CH ₂), 3.80(s,H,2CH ₃), 4.4(s,H,NH ₂), 7.5-8.0(m,H,Ar-H),					
<i>C</i> 4	2.08(s,H,2CH ₃), 2.50(s,H,CH ₂), 3.63(s,H,CH ₂), 3.29(s,H,2CH ₃), 7.2-7.8(m,H,Ar-H),11(s,H,OH), 4.3 (N=C-H).					
C5	2.48(s,H,2CH3), 2.6(s,H,CH2), 3.18(s,H,CH2), 6.8(m,H,Ar-H), 10.20 (s,H,OH), 4.4 (N=C-H).					
<i>C</i> 6	2.2(s,H,2CH ₃), 2.5(s,H,CH ₂), 3.00(s,H,CH ₂), 7.4(m,H,Ar-H), 11.09 (s,1H, OH), 4.5(N=C-H), 4.43 (H,Ar-OH).					
<i>C</i> ₇	2.05(s,H,2CH ₃), 3.53(s,H,4CH ₃), 2.48(s,H,CH ₂), 2.94(s,H,CH ₂), 3.72(s,H,CH ₂), 3.90(s,H,CH), 11.12(s,H,OH), 7.0-7.5(m,H,Ar-H).					
<i>C</i> 8	2.15(s,H,2CH ₃), 2.5(s,H,CH ₂), 2.98(s,H,CH), 3.2(s,H2CH ₃), 3.4(s,H,CH ₂), 3.5(s,H.CH), 7.4-7.9(m,H,Ar-H), 13.21(s,H,OH).					
<i>C</i> 9	2.07(s,H,2CH ₃), 2.79(s,H,CH ₂), 2.52(s,H,CH ₂), 3.05(s,H,2CH ₃), 3.24(s,H,CH ₂), 3.5(s,H,CH), 7.7-8.0(m,H,Ar-H), 11.28 (s,1H,OH), 4.30(H,Ar-OH).					





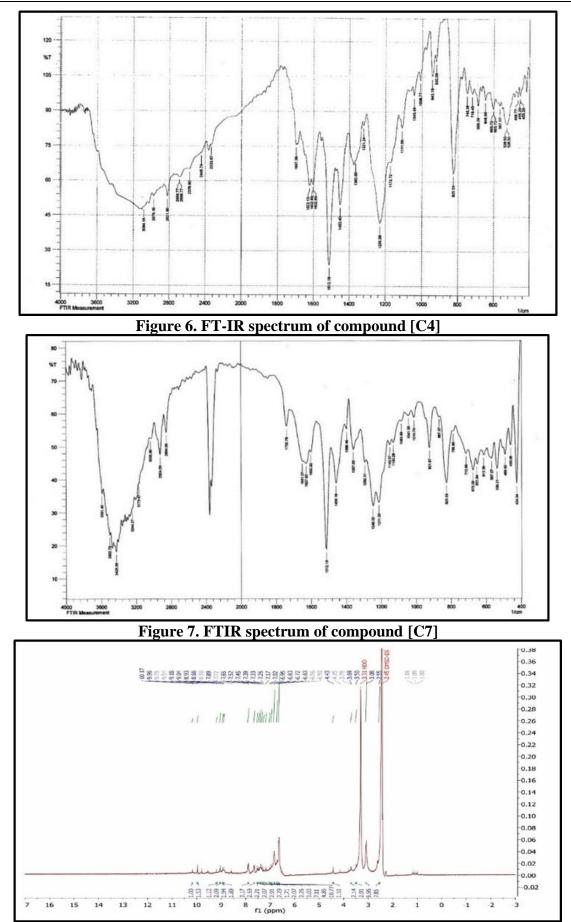


Figure 8. FT-IR spectrum of compound [C8]

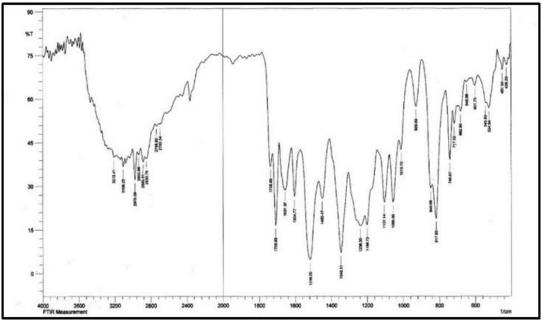


Figure 9. 1H-NMR spectroscopy of the compound [C5]

Biological Activity ^(14,15) The comps [C1-C5] biological activity was tested towards the five types of Gram-positive and Gram-negative bacteria, where **Table 4. Biological activi**

Dimethylsulphoxid (DMSO) was considered a control. The results of the comps [C1-C5] and control were shown in Table 4 and Figure 10.

Fable 4. Biological	activity of the	compounds [C1-C5]
abie 4. Diviogical	activity of the	

Comp No:	E. coli	Psuedomonas aeruginosa	S. aurous	Streptococcus pyogenes	Candida albicans
1	5 mm	10 mm		<u>10 mm</u>	
2	5 mm				
3	5 mm	13 mm	5 mm		10 mm
4	10 mm		10 mm	10 mm	
5		14 mm	5 mm		
DMSO	0	0	0	0	0

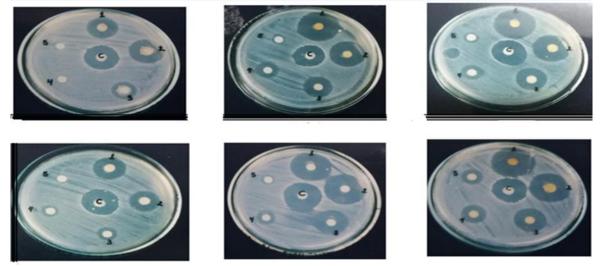


Figure 10. Biological activity of compounds [C1-C5]

CONCLUSION

In this paper, several silver oxide nanoparticles were synthesized from the locally available plant "Ginger" (*Zingiber officinale*) with high products and used as a catalyst in preparing the essential pyran derivatives industrially. These compounds contain many active groups that can be cyclized and involved in several industrially important and high product reactions.

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