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Synthesis of Some Organic Hydrazine Compounds Using Microwave Radiation

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Abstract

This work was included the preparation of substituted organic hydrazine compounds from acid hydrazide (succinic dihydrazide and malonoicdihydrazide) through the reaction with different aromatic aldehyde compounds using microwave radiation. This study was also identified the product compounds by measuring its physical constants (melting point) and measuring infrared spectra. Confirmation for the structures of the prepared compounds by using infrared Spectro and measuring some physical constants, that sure the reactions were complete by using new energy radiation under green chemistry which accelerated the reaction in a few seconds and without byproduct.

Keywords: Synthesis; Hydrazine; Characterization and microwave radiation

Introduction

Hydrazones are considered important organic compounds that have the following structure (R1R2C=N-NH2) and which are cleaved from aldehydes and ketones [1], where the oxygen has been replaced by the functional or active group (N-NH2) and usually prepared through the reaction between hydrazine with aldehydes or ketones [2]. Extensive research has been carried out to prepare derivatives of hydrazine, especially acyl –hydrazone, which has biological properties and efficacy and also has application in analytical fields. therefore, it is used as a catalyst in chemical reactions [3]. Hydrazone is complexed with minerals, which are characterized by strong activity towards properties [4]. Pharmaceutically, the cyclic products of acylhydraseone compounds are important heterocyclic compounds with a wide range of biological and pharmaceutical activities [5]. A group of researchers was also able to prepare a group of hydrazones with aldehydes and ketones using the microwave [6]. The efficacy of the prepared compounds was measured against microbes [7]. A group of researchers was able to prepare hydrazones that contain long chains of domesticated hydrocarbons that are effectively used as agricultural pesticides using the

microwave *(8)*. This work was aimed at the synthesis of some organic hydrazine compounds using microwave radiation.

Materials and Methods

The infrared spectrum was measured by a device (FT.I.R.470, infrared Spectro - Shimadzu) and measure the degree of melting with a device (Elecrtrotheromal (M.P.) Gallen Kamp). Then conduct the reactions using a microwave type device Microwellengrat 8020 – (Privilege).

Procedure: 1,8 –Bis (substituted) succinic dihydrazid ($N_{12} - N_1$)

Mixing (0.001 mol) of acid hydrazides (Succinic dihydrazide) and (0.002 mol) of various solid aromatic aldehydrate compensators were mixed in a ceramic mortar and crushed well. If it is a liquid, mix it by means of a vibrator in a beaker volume of (50 ml), then add (4-6 drops) of dimethyl formaldehyde and put the baker in an oven. Microwave and irradiate the mixture as indicated in Tables 1 and 2 for time and microwave energy. After the end of microwave energy and reaction, an oily substance was obtained and the substance treated by adding ice water with continuous stirring in order to convert the oily substance into a precipitated solid. In some cases, the precipitate was washed by a 9:1 mixture of (ethyl acetoacetate. Diethyl ether) and the solid filtered. And washed it with cold water several times and done crystallization using absolute ethanol.

The physical properties of these compounds were shown in Table 1-2 and Figure 1 with the general formula for this type of compounds and Figure 2 shown the general formula for the type of vehicle.

Ar-CH=NH-CO-CH_2 CH_2-CO-NH-N=CH-Ar

Figure 1: The structural form of hydrazones.

($N_{25} - N_{13}$)1,7-Bis(substituted)malonoic dihydrzide

In the same way, (N13-N25) compounds were prepared using malonicdihydrazide. The physical properties of these compounds were shown in Table 1and 2

Ar-CH=NH-CO-CH_2-CO-NH-N=CH-Ar

Figure 2: The general formula for this type of vehicle.

Table 1: The physical constants for the prepared hydrazones (N_1-N_{11}).

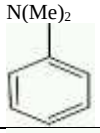
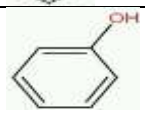
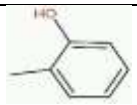
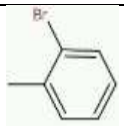
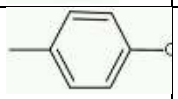
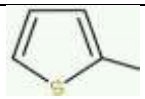
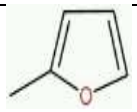
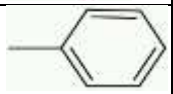
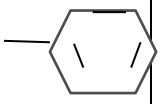
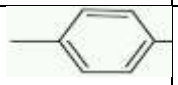
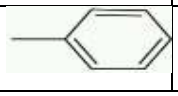
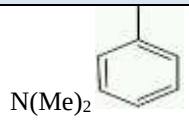
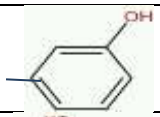
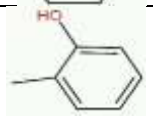
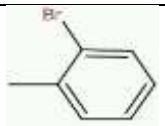
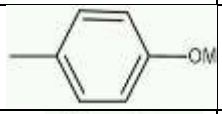
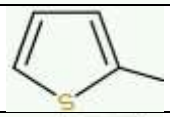
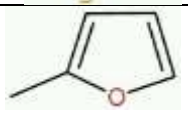
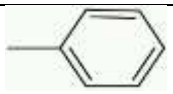
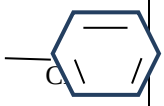
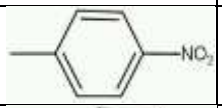
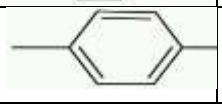
Comp. NO	Ar	Color	Yield (%)	m.p.c	MwI Power watt	RT (min)
N1	$N(Me)_2$ 	Yellow	80	216-214	80	1.5
N2		Yellow	78	216-218	180	2.0
N3		Yellow	90	240-242	360	1.5
N4		Yellow	95	213-222	80	2.0
N5		Yellow	95	213-215	80	1.3
N6		Light Brown	95	228-230	80	1.2
N7		Brown	70	220-222	80	1.5
N8		Yellow	85	207-209	380	2.0
N9		Yellow	78	223-225	180	2.0
N10		Light Yellow	75	226-268	80	2.0
N11		Light Yellow	95	206-208	80	2.0

Table 2: The physical constants for the prepared hydrazones (N1b3 - N23)

Comp. NO	Ar	Color	Yield (%)	m.p.c	MwI Power Watt	Reaction Time-Min
N13		Yellow	80	232-230	80	0.
N14		White Yellow	75	217-214	80	1.3
N15		White Yellow	80	246-244	80	1.5
N16		Light Yellow	80	219-217	80	2.0
N17		White Yellow	95	210-208	80	1.3
N18		Light Brown	95	197-196	80	1.2
N19		Yellow Brown	70	232-230	80	1.5
N20		Light Yellow	80	221-219	80	1.3
N21		White	78	213-212	180	2.0
N22		Light Yellow	75	272-270	80	2.0
N23		Yellow	95	228-226	80	2.0

Results and Discussion

These compounds were prepared from the reaction of the acid hydrazides prepared in the first stage with a mullein of aromatic aldehydes when DMF was present as a solvent and by using microwave device. It

involves crushing the reactants together and then adding several drops of DMF, which are then irradiated in the microwave to form the hydrazine product as shown in the diagram. The reaction takes place here with the mechanism of nucleophilic addition and deletion as a result of the attack of the nitrogen atom that contains a pair of non-participating electrons on the carbon atom in the in the carbonyl aldehyde group, which leads to a new bonding euphoria and the formation of an unstable intermediate e compound by losing a water molecule that forms hydrazine. If it is considered more stable than the intermediate compound as a result of the cascade resonance between the double bond formed **(–C=N-)** and the monds in the benzene ring ,as shown in the diagram .When diagnosing the prepared hydrazine compound s as in Figures 1 and 2 for the compounds (**N_1-N_{25}**) with the infrared spectrum , it was observed that a beam of medium intensity **(N-H)** stretched between the two regions **(3250-3183)** cm-1 was observed and a clear band was observed due to the stretching of the carbonyl group of hydrazine . It was within the **(1695-1650) cm-1** as the disappearance of the beam was observed the carbonyl stretch that belongs to the aldehyde ranges between **(1735-1715) cm-1,** while a medium –strength bundle stretching of the double bundle **(C=N),** was observed due to the double bond stretching within the region **(1690-1580) cm-1**. Also, medium severity bundle was observed due to the double bundle stretching **(C=C)** of the aromatic ring, and it was within the range **(1600-1510) cm**-1, as in Figures 1 and 2**,** Tables 3 and 4 were **s**hown the results of the infrared absorption spectrum. Figures 3-5 were shown the infrared spectra of compounds prepared from hydrazine as following Reaction and mechanical equation:

These compounds were prepared from the reaction of the acid hydrazides prepared in the first stage with mullein of aromatic aldehydes when DMF was present as a solvent and by using a microwave device. It involves crushing the reactants together and then adding several drops of DMF, which are then irradiated in the microwave to form the hydrazine product as shown in the diagram. The reaction takes place here with the mechanism of nucleophilic addition and deletion as a result of the attack of the nitrogen atom that contains a pair of non-participating electrons on the carbon atom in the carbonyl aldehyde group, which leads to a new bonding euphoria and the formation of an unstable intermediate e compound by losing a water molecule that forms hydrazine. If it is considered more stable than the intermediate compound as a result of the cascade resonance between the double bond formed **(–C=N-)** and the minds in the benzene ring, as shown in the diagram. When diagnosing the prepared hydrazine compound s as in Figures 1 and 2 for the compounds **(N1-N25)** with the infrared spectrum, it was observed that a beam of medium intensity **(N-H)** stretched between the two regions **(3250-3183)** cm-1 was observed and a clear band was observed due to the stretching of the carbonyl group of hydrazine. It was within the **(1695-1650)** cm-1 as the disappearance of the beam was observed the carbonyl stretch that belongs to the aldehyde ranges between **(1735-1715)** cm-1, while a medium-strength bundle stretching of the double-bundle **(C=N)**, was observed due to the double bond stretching within the region **(1690-1580)** cm-1. Also, a medium severity bundle was observed due to the double-bundle stretching **(C=C)** of the aromatic ring, and it was within the range **(1600-1510)** cm-1, as in Figures 1 and 2, Tables 3 and 4 were shown the results of the infrared absorption spectrum. Figures 3-5 were shown the infrared spectra of compounds prepared from hydrazine as following Reaction and mechanical equation:

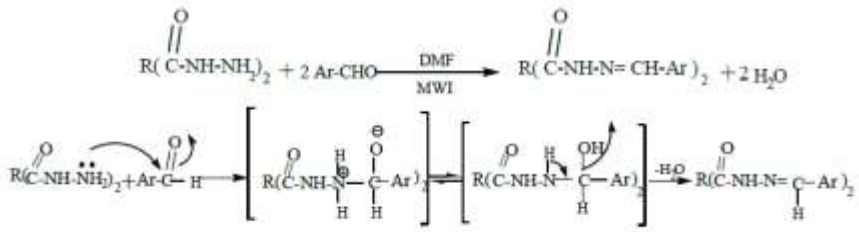


Table 3: Infrared spectrum (IR) results of the prepared hyrazones.

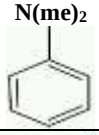
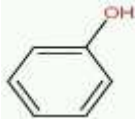
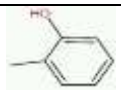
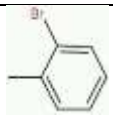
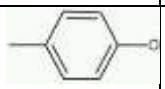
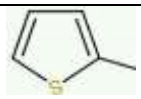
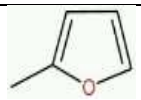
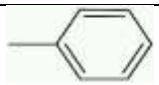
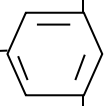
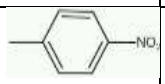
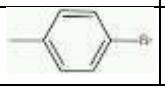
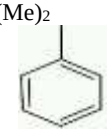
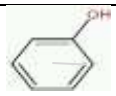
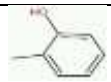
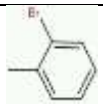
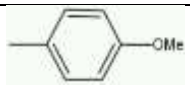
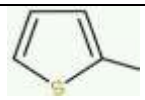
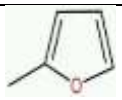
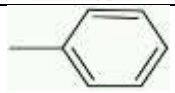
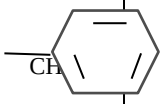
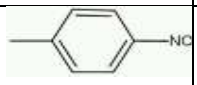
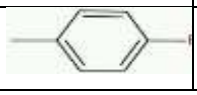
Comp. NO	Ar	l.R. KBr_disc v(cm^{-1}				
		Aratic c=c	C = N	Hydrazone c=o	NH	Others
N1	$N(me)_2$ 	1523	1602	1655	3211	
N2		1580	1605	1670	3200	3450 OH_
N3		1560	1600	1695	3250	3400 OH_
N4		1540	1650	1690	3200	840 C_Br
N5		1546	1569	1651	3206	1270 C_O_C
N6		1558	1595	1651	3204	
N7		1559	1610	1670	3200	1274 C_O_C
N8		1559	1607	1680	3214	
N9	CH=CH 	1565	1590	1650	3209	
N10		1540	1615	1690	3210	1518asy (NO_2) 1345asy(NO_2)
N11		1590	1606	1651	3196	850 C_Br

Table 4: Infrared spectrum (IR) results of the prepared hyrazones

Comp NO	Ar	l.R. KBr_disc v(cm^{-1}				
		Aratic c=c	c=N	Hydrazone c=o	NH	Others
N13	$N(Me)_2$ 	1600	1635	1673	3183	
N14		1560	1600	1660	3208	3430 OH_
N15		1540	1590	1690	3230	3400 OH_
N16		1545	1650	1670	3190	860 C-Br
N17		1546	1569	1651	3206	1271 C_O_C
N18		1560	1590	1670	3200	
N19		1546	1569	1651	3206	1270 C_O_C
N20		1550	1590	1670	3200	
N21		1560	1585	1670	3190	
N22		1560	1600	1680	3220	1520 Asy (NO_2)
N23		1580	1600	1660	3208	810 C-Br-

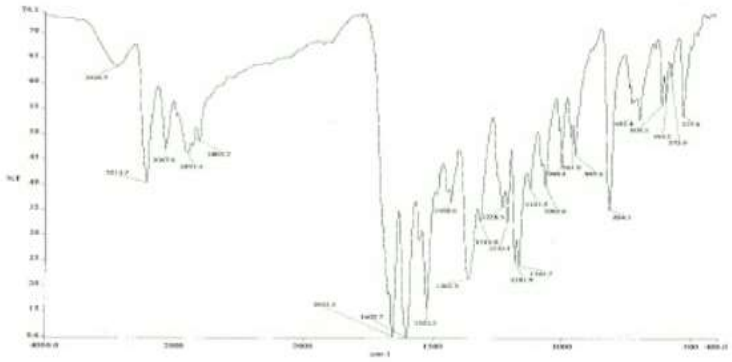


Figure 3: The infrared spectrum of the compound (N1).

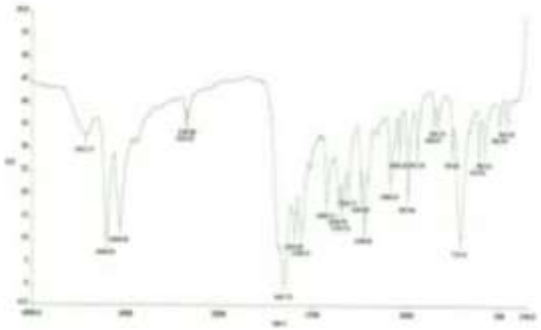


Figure 4: The infrared spectrum of the compound (N6).

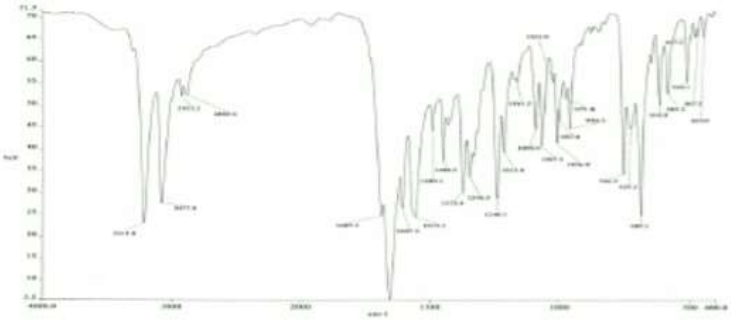


Figure 5: The infrared spectrum of the compound (N8).

Conclusion

This was used in the search of the microwave radiation successfully in the preparation of organic hydrants compound without suburban materials, no secondary outputs and dangerous waste .so clean the pavement "Green chemistry" and the use of this radiation is a time of interaction from 2-4 minutes and the product of 80-90 % reverse traditional heating methods where it takes time of interaction from 2-3 hours and the heating of the microwave reduces to the amount of reactions and enhances its terms by reducing side interactions.

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