

Inventing of Macrocyclic Formazan Compounds with Their Evaluation in Nano- Behavior in the Scanning Microscope and Chromatography

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ABSTRACT

Cyclic Formazan was invented and prepared for the first time by researcher Dr. Nagham Aljamali was established in April 2021. Therefore, it is considered one of the developed and modern compounds, which is described by a lack of references and researches. Therefore, the researcher, Dr. Nagham prepared and created a series of these compounds and studied several applications, including biological, pharmaceutical studies, also as antifungal, antibacterial, and anticancer agents, and here in this research, cyclic formazan was studied as medical nanomaterials. A number of technical spectroscopic studies have been used to demonstrate their chemical structures which provided clear evidences of their chemical structures via many technical instruments like (FT IR-Spectra , 1H.NMR-Spectra, Mass-Spectra), Melting points, other studies represented by evolution them as Nano-compounds by {Scanning Electron Microscopy (FESEM), and their Chromatographic behavior}.

Keywords: Cyclic Formazan; Formazan; Imine; Schiff Base; Azo; Aldamine; Anil; Scanning Electron Microscopy (FESEM); Chromatography

Introduction

Cyclo- Formazan is one of the modern compounds in the field of organic chemistry and is considered an innovation by Dr. Nagham Aljamali in April 2021 when it was prepared for the first time globally [1,2]. And because their studies and references are a few for this cause the researcher Dr. Nagham Aljamali prepared and carried out various compounds from Macrocyclic-Formazan by using various conditions and different basic medium [3-7] like (Pyridine ,Pipridine ,5 % Sodium hydroxide, Triethyl amine,...) [3], and linked them with heterocyclic compounds and other compounds with more than two hetero atoms to increase their effectiveness [7-11] , biological [13,16] and industrial applications [17,20]. Cyclic Formazan has cyclic structure of (-N=N=C-N- in cyclic structure) or (-N=N-C-N-NH- in cyclic structure) according

to type of amine in reaction [1,2],. They were considered among the organic compounds of importance in organic chemistry because they contain two highly effective groups in several fields of chemistry [1,21-24], especially in coordination chemistry [25-27], as a ligands because they contain free electrons of donor atoms to coordinate with ions to form complexes [28-30]. Formazan also tested in many studies as anti-bacterial [31-34] and anti-fungal compounds [35-36] and types of cancer [37-39], especially breast [40] and laryngeal cancers [41], as anti-bacterial [42] and anti-fungal [43,44], and other studies [40-44].

Instruments and Experimental Part

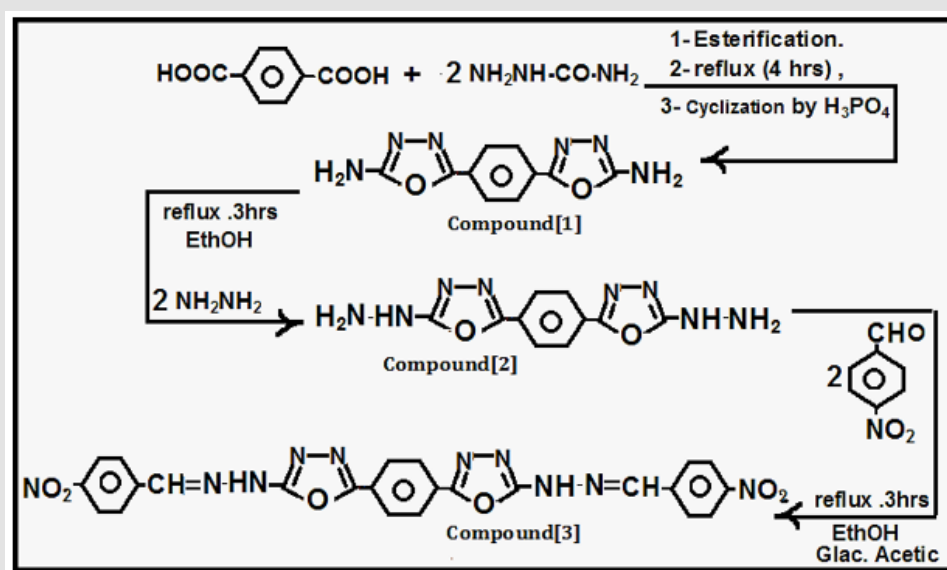
All melting points were uncorrected and dignified on an

electro-thermal apparatus (Switzerland) in an open capillary tube. FTIR spectra were detailed on Fourier transform infrared spectrometer (FT-IR) in (FT-IR- 3600) infrared spectrometer via employing KBr Pellet technique., ¹H.NMR spectra were recorded in DMSO-d₆ as solvent using (TMS) as internal standard and chemical shifts are expressed as (δ ppm), also Mass- Spectra for some of them other studies represented by evolution them as Nano-compounds by {Scanning Electron Microscopy (FESEM), and their Chromatographic behavior}.

Procedures

Preparation of Compounds {1, 2, 3}: Terephthalic acid (0.01 mole) was dissolved in (30 ml) absolute ethanol (2ml) of Sulfuric

acid with refluxing for (2 hrs) in esterification step, then the ester will cyclize with semicarbazide (0.02 mole) with refluxing for (4 hrs) in presence of phosphoric acid as closing agent in cyclization step, according to procedures [4-7], the product filtered, dried, recrystallized to yield Ox diazole amine Compound [1], which reacted (0.01 mole) with (0.02 mole) hydrazine in refluxing step for (3 hrs) according to procedures [4-7], the product filtered ,dried, recrystallized to yield Oxadiazole hydrazine Compound [2], which refluxed (0.01 mole) with (0.02 mole) of p-nitrobenzaldehyde for (3 hrs) in presence of (3 drops of glacial acetic acid), according to procedure [4-7], the product filtered ,dried ,recrystallized to yield Imine -Compound [3] (Scheme 1).

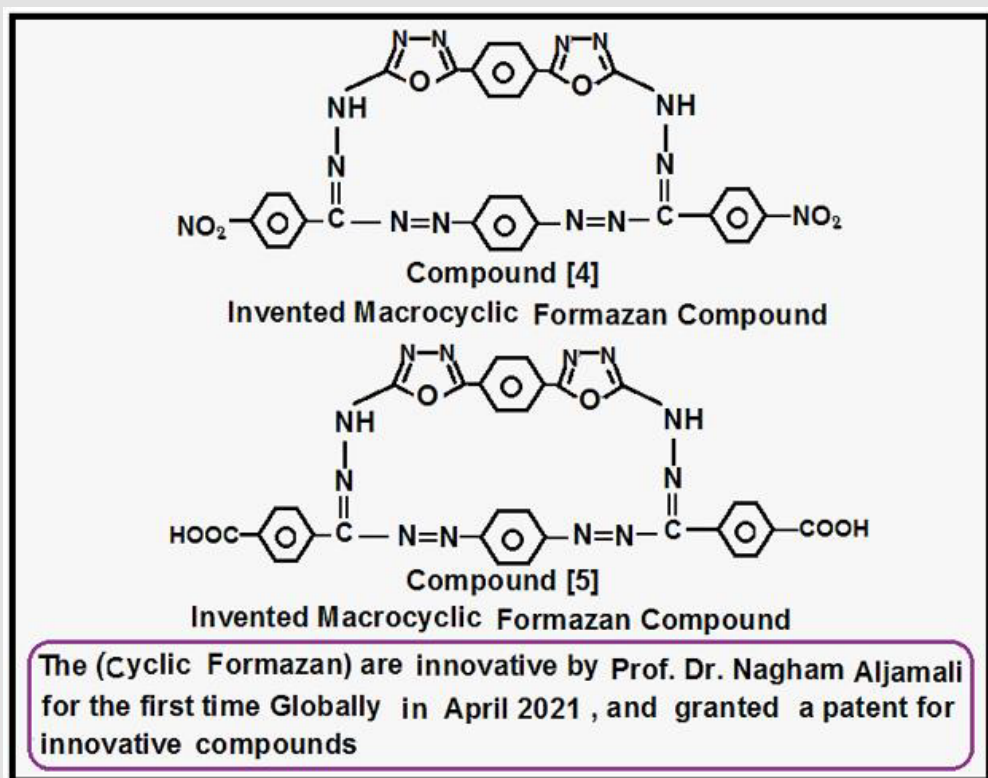


Scheme 1: Synthesis of Compounds {1, 2, 3}.

Creation of Inventive Macrocylic Formazan Compound {4}: Compound [3] was (0.01 mole) reacted in presence of (Pyridine) with (0.01 mole) of diazo salt of p- phenyl diamine via many steps in basic medium to formation Invented Macrocylic Formazan after (15 hrs), the product filtered, dried, washed by distilled water, recrystallized to yield Invented Macrocylic Formazan [4] by following literatures [1,2].

Creation of Inventive Macrocylic Formazan Compound {5}: Compound [2] refluxed (0.01 mole with (0.02 mole) of p-formal

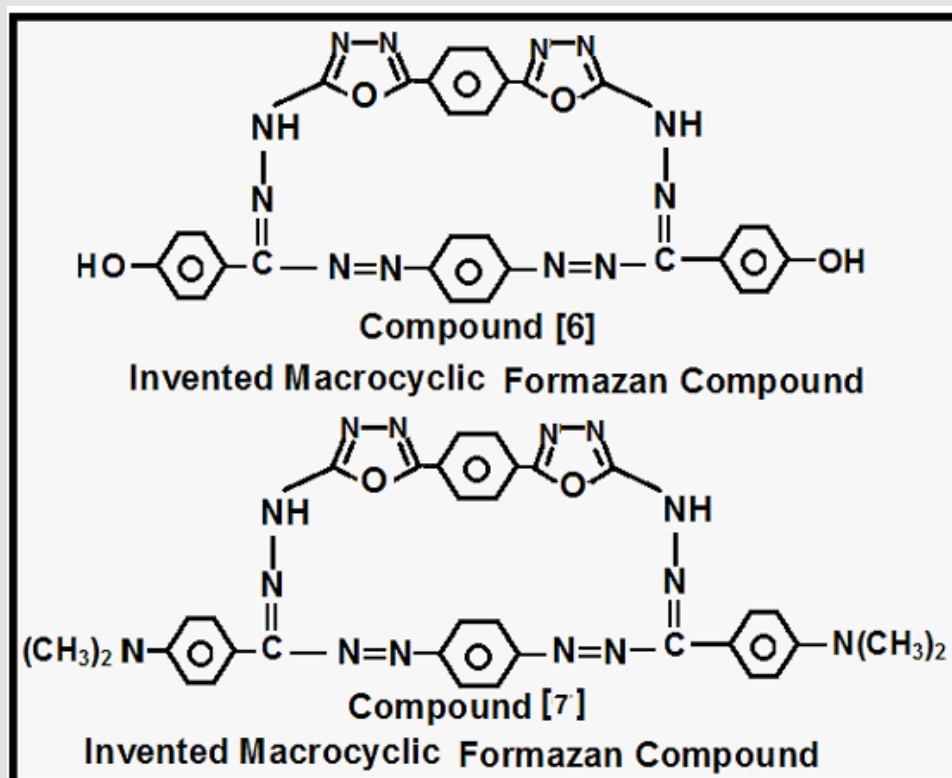
benzoic acid in presence of (2-3 drops) of glacial acetic acid for (2 hrs) in absolute ethanol according to procedure [4-7], the product filtered ,dried ,recrystallized to yield Imine -Compound that (0.01 mole) was reacted in presence [1-3] of (Triethyl amine) with (0.01 mole) of diazo salt of p- phenyl diamine via many steps in basic medium to formation Invented Macrocylic Formazan after (12 hrs), the product filtered, dried, washed by distilled water, recrystallized to yield Invented Macrocylic Formazan [5] by following literatures [1,2] (Scheme 2).



Scheme 2: Creation of Invented Macrocyclic Formazan Compounds {4, 5}.

Creation of Inventive Macrocyclic Formazan Compound{6}: Compound [2] refluxed (0.01 mole with (0.02 mole) of p-formal phenol in presence of (2-3 drops) of glacial acetic acid for (3 hrs) in absolute ethanol according to procedure [3-7], the product filtered ,dried ,recrystallized to yield Imine -Compound that (0.01 mole) was reacted in presence [1,2] of (Pipyridine) with (0.01 mole) of diazo salt of p- phenyl diamine via many steps in basic medium to formation Invented Macrocyclic Formazan after (10 hrs), the product filtered ,dried ,washed by distilled water, recrystallized to yield Invented Macrocyclic Formazan [6] by following literatures [1,2].

Creation of Inventive Macrocyclic Formazan Compound{7}: Compound [2] refluxed (0.01 mole with (0.02 mole) of 4-N,N-dimethylamine benzaldehyde in presence of (2-3 drops) of glacial acetic acid for (2 hrs) in absolute ethanol according to procedure[3-7], the product filtered ,dried ,recrystallized to yield Imine -Compound that (0.01 mole) was reacted in presence [1-3] of (5 % NaOH) with (0.01 mole) of diazo salt of p- phenyl diamine via many steps in basic medium to formation Invented Macrocyclic Formazan after (20 hrs), the product filtered, dried, washed by distilled water, recrystallized to yield Invented Macrocyclic Formazan [7] by following literatures [1,2] (Scheme 3).



Scheme 3: Creation of Invented Macrocylic Formazan Compounds {6, 7}.

Results and Discussion

In recently scientific paper, various of Invented Macrocylic Formazan Compounds have been created in same procedure that followed and invented [1,2] by Dr. Nagham in April 2021 that got a patent to invention of Macrocylic Formazan compounds, then several studies were carried out to improve these innovative compounds by the using of spectral identification like : ¹H.NMR spectra, FT.IR- Spectra, Mass- Spectra., other studies represented by (Melting points, other studies represented by evolution them as Nano-compounds via {Scanning Electron Microscopy (FESEM) ,and their Chromatographic behavior}., all the results are shown in tables and figures.

Spectral Investigation

FT.IR- Spectral Indication of Invented Macrocylic Formazan Compounds: The first characterization of innovative compounds by shifting of frequencies of Imine group (CH=N) in starting compounds (Imine compounds) that were about at (1615 , 1610, 1618, 1620) Cm^{-1} respectively in all starting materials of imine compounds that were shifted to (1630 , 1627 , 1631, 1642) Cm^{-1} for (-C=N-) due to formation of Macrocylic Formazan,

also appearance of three bands due to partitions of azo group of Formazan in Macrocycle (-N=N-) are (1429 ,1451, 1476) Cm^{-1} for (-N=N-C-) in compound {4}., and other compound like this., all frequencies clarified according to reference [1,33].

¹H.NMR- Spectral Indication of Invented Macrocylic Formazan Compounds: The second characterization of innovative compounds by disappearance of peak for imine group (CH=N) in starting compound (Imine compound) that were at δ (8.13) in Compound {3} (starting compound) due to formation of (N=C-N=N) for (Formazan groups) in innovated compounds [4, 5, 6, 7], also in compound [5] appeared peak at δ (12.31) due to proton of carboxyl group (COOH), while compound [6] appeared peak at δ (10.82) due to proton of hydroxyl group (OH), all peaks explained according to reference [33].

Mass- Spectral Indication of Invented Macrocylic Formazan Compounds: The third characterization of inventive compounds by partition of innovative cyclic compounds via appearance of fragments in spectra in (Figures 1 & 2).

Other Characterization: All Invented Macrocylic Formazan derivatives were studied to collect all the chemical and physical properties, in (Table 1).

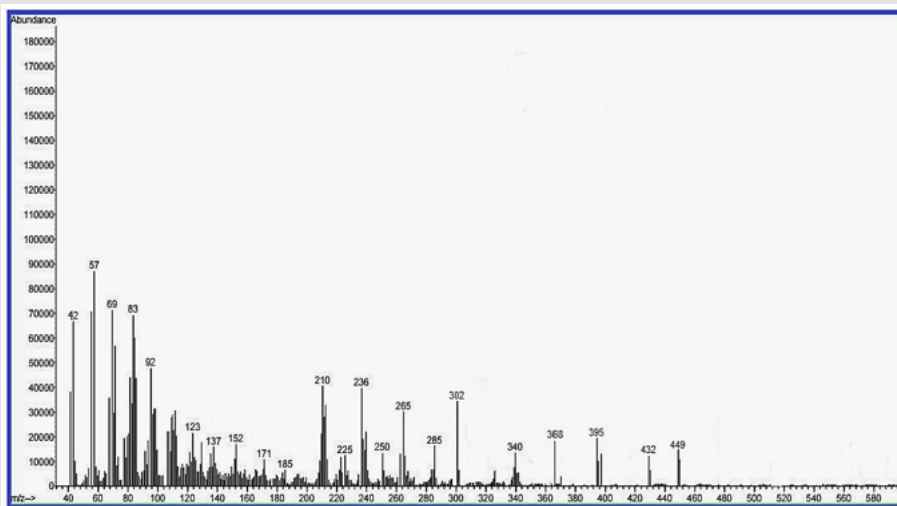


Figure 1: Mass-Spectrum of Invented Macrocyclic Formazan Compound [4].

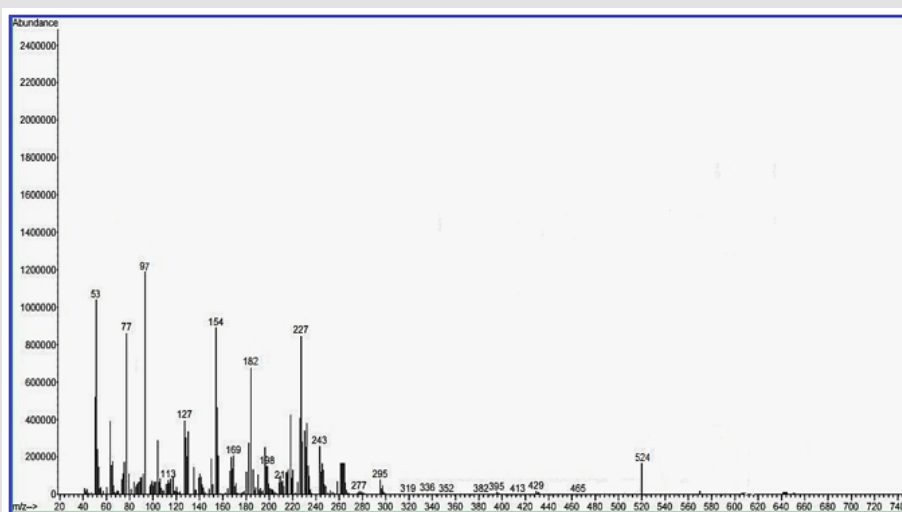


Figure 2: Mass-Spectrum of Invented Macrocyclic Formazan Compound [7].

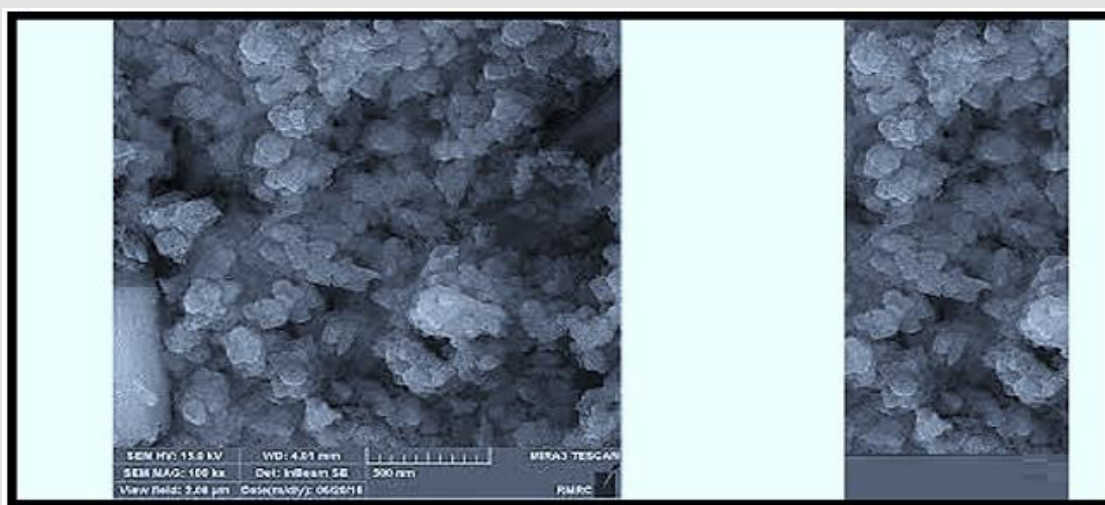


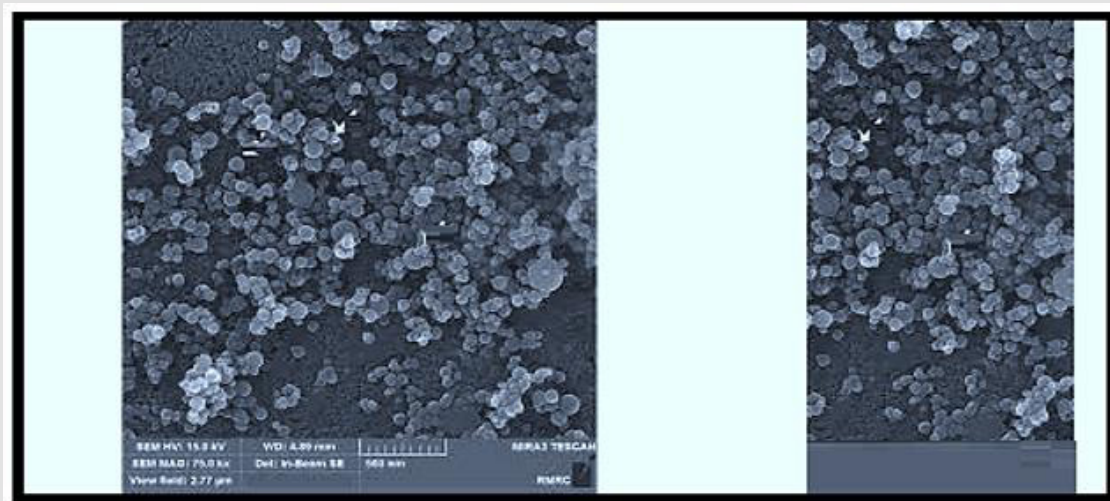
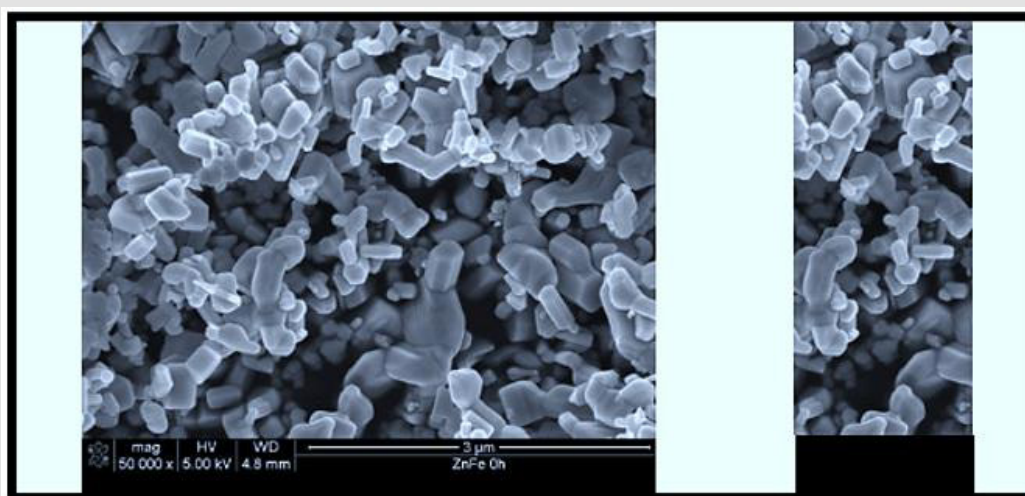
Figure 3: Scanning Electron Microscopy of Invented Cyclic Formazan [4].

Table 1: Other characterization of Invented Macrocylic Formazan Compounds.

Innovated Comps.	P %	Color	M .P C°	Rf	Solvents (TLC)
Innovated Comp.{1}	76	Bill Yellow	160	0.62	Ethanol : Dioxan
Innovated Comp.{2}	76	Deep Yellow	184	0.56	Ethanol : Dioxan
Innovated Comp.{3}	74	Yellowish Orange	198	0.6	Ethanol : Dioxan
Innovated Comp.{4}	86	Reddish Yellow	214	0.66	Ethanol : Dioxan
Innovated Comp.{5}	82	Orange	226	0.58	Ethanol : Dioxan
Innovated Comp.{6}	86	Reddish Orange	220	0.6	Ethanol : Dioxan
Innovated Comp.{7}	80	Orange	228	0.62	Ethanol : Dioxan

Scanning Electron Microscopy (FESEM): Scanning Electron Microscopy (FESEM) of the Innovated Cyclic Formazan compounds (for morphological properties) that revealed in this research that they have a spherical shape and have granular sizes within the Nano-scale they have an average size of (44. 23 , 40. 12 , 46. 61, 55. 97) nanometers for Cyclic Formazan Compounds [4, 5, 6, 7]

respectively, so the surface area increases and this characteristic makes it eligible for medical uses because it has a small granular size , spherical shape within the nano-scale that is used in medical fields as a treatment for many types of cancers as well as in the industrial field, (Figures 3-6).

**Figure 4:** Scanning Electron Microscopy of Invented Cyclic Formazan [5].**Figure 5:** Scanning Electron Microscopy of Invented Cyclic Formazan [6].

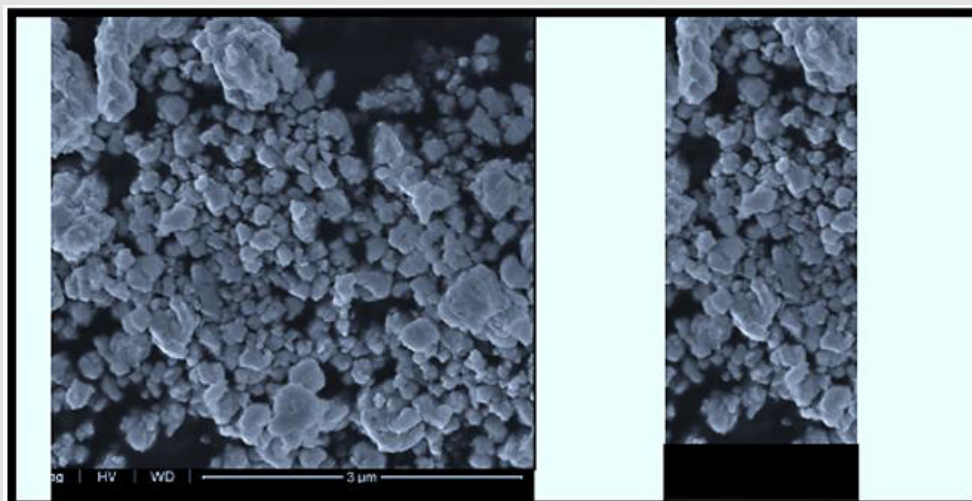


Figure 6: Scanning Electron Microscopy of Invented Cyclic Formazan [7].

Chromatographic Study for Invented Cyclic Formazan Compounds: This section of the study involved a study of the chromatographic separation of the invented cyclic formazan compounds to know the effect of the effective groups in the chemical composition on the separation according to procedures [39-43], such as polar groups. In this work, Cyclic Formazan Compound {5} is the slowest compound in the separation because it contains

two polar carboxyl groups (OH) that are affected when descending during the season and followed by Cyclic Formazan compound {6} according to polarity, then compound [4], and last one is compound [7], for this reason the compound [7] separated faster than other compounds due to its structure is less polarity and less interaction with column, (Figures 7-10).

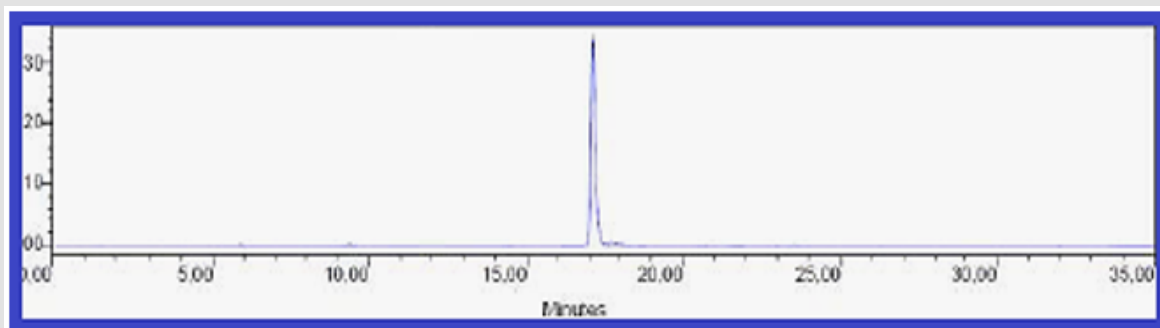


Figure 7: Chromatogram of Invented Cyclic Formazan Compound [4].

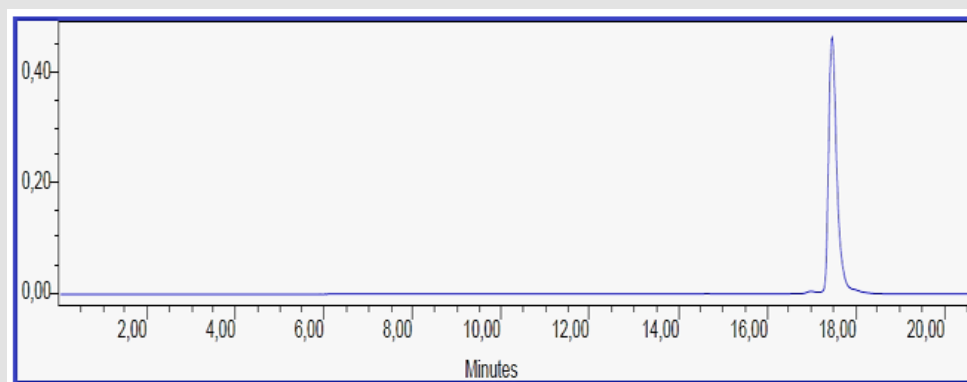


Figure 8: Chromatogram of Invented Cyclic Formazan Compound [5].

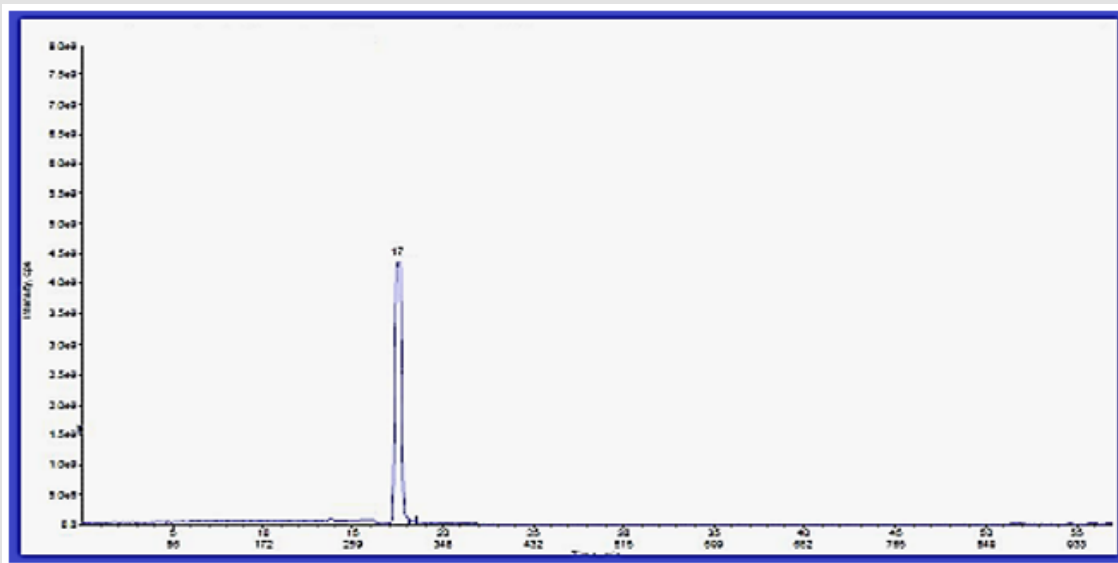


Figure 9: Chromatogram of Invented Cyclic Formazan Compound [6].

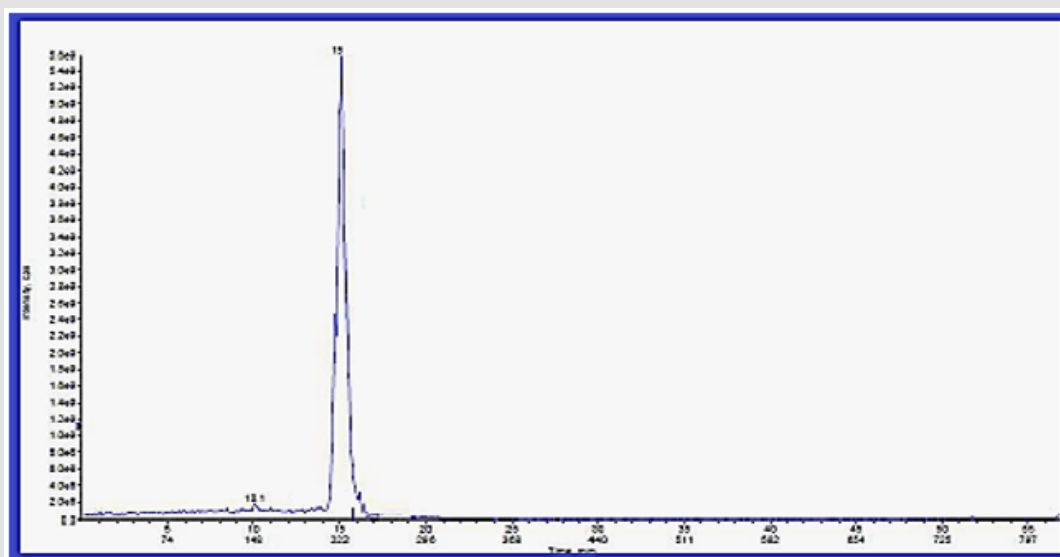


Figure 10: Chromatogram of Invented Cyclic Formazan Compounds [7].

Conclusion

All Invented Macrocylic Formazan compounds gave good evidences for their structures via various spectral techniques, also some of them studied like scanning microscope appeared Nano-properties for these compounds, which means that the cyclic Formazan compounds can be good drug delivery to treatment and medical applications.

Conflict of Interest

The authors declare that there is no conflict of interest.

Funding Source

None.

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