Synthesis and Characterization of some New 3,3'-(1,4-Phenylene) Bis (1-(4-Aminophenyl) Prop-2-en-1-one) Amide Derivatives

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Doi:10.29072/basjs.20220214

ARTICLE INFO

ABSTRACT

Keywords

Bis-chalcone,
Condensation, Amide derivatives,
Carboxylic acid,
Acrylamide.

A new amide derivative series [A1-A8] was prepared in good yield by direct reflux condensation of bis-chalcone [3,3'-(1,4-phenylene) bis (1-(4-aminophenyl) prop-2-en-1-one)] with different carboxylic acids and acrylamide in the presence of a suitable solvent and amount of sodium hydroxide (NaOH). The structures of the synthesized amide were characterized by various spectral techniques, including FTIR (Fourier-Transform Infrared Spectroscopy), 1H-NMR (Proton Nuclear Magnetic Resonance Spectroscopy), and mass spectroscopy, and the resultant spectra confirmed the expected structure of the prepared bis-chalcone derivative.

Received 15 Jan 2022; Received in revised form 23 Jun 2022; Accepted 3 July 2022,Published 31 Aug 2022

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1. Introduction

Amide compounds are a very important category of organic compounds, so the formation of amide compounds has attracted significant interest due to their importance in organic and bioorganic chemistry, their value as intermediates in organic synthesis, and a wide range of applications in the chemical industry [1-3]. Because of the amide functionality's apparent importance, by far the most popular procedure is condensation between a carboxylic acid and an amine [5]. As environmentally-friendly alternatives to standard chemical transformations are sought, the use of condensing and activating agents is becoming increasingly unpopular [6]. To achieve that end, the most desirable path to construct an amide bond would be the direct condensation of an amine with a carboxylic acid. Nevertheless, even though direct amide formation was reported as early as 1858, there have been comparatively few reports in the literature referring to direct condensation, and it remains an understudied and misunderstood interaction mechanism [7]. The amide linkages appear as an important structural component in peptides, many natural polymeric products, and pharmaceuticals. They are not only the primary chemical links between proteins and pharmaceuticals, but they are also the foundation for some of the most commonly used synthetic polymers [1,4,8]. Heating a mixture of amine and an acid, Jursic, and Zdravkovski [9] have elucidated as the method of choice for the direct synthesis of many amides. The yields depend on the physical properties and thermal stabilization of the starting materials and range from good to excellent. The idealistic starting materials should have melting points below 200 °C, should not be highly volatile, and should be thermally stable at that temperature for 30 minutes. The method is characterized, among others, by its simplicity, low cost, and short reaction time. It can also be implemented on a very large scale and does not require solvent and special purification of the starting materials. Chhatwa et al. [10] have described a new method for the direct synthesis of primary and secondary amides from carboxylic acids using Mg (NO3)2.6 H2O or imidazole as a cheap and readily available catalyst, and urea as a stable and easy-to-manipulate nitrogen source. This methodology is especially advantageous for the direct synthesis of primary and methyl amides, avoiding the use of ammonia and methylamine gas. Furthermore, the transformation does not demand the employment of coupling or activating agents, which are commonly required. Fu et al. [11] have carried out the direct synthesis of amides from amines and carboxylic acids under hydrothermal conditions. They have found that several amides (12 examples) are easily prepared during a direct condensation between amines and carboxylic acids, with an amide yield of up to 90% over a schedule of hours. Hydrothermal experiments have been carried out to obtain apparent

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rate constants for amide synthesis through sequential periods. In this work, the reaction of bis-chalcone with two series of carboxylic acids was carried out to synthesize a new long-chain amide molecule. The first chain of mono-carboxylic acids and the second chain of di-carboxylic acids were synthesized via direct reaction. This methodology is particularly useful for the direct synthesis of secondary amides from carboxylic acids. Furthermore, the alteration does not require the employment of coupling or activating agents, which are commonly required.

2. Experimental

2.1 Materials

All three Sigma-Aldrich, Merck, and Fluka companies supplied all the chemicals and solvents used in this study. The uncorrected melting points of the prepared compounds were recorded on an open capillary status thermal point apparatus (England). The FTIR spectra of all synthesized compounds were measured as KBr discs in the region of 4000-400 cm^{-1} by using Shimadzu FTIR-8400 (Japan). The 1H-NMR spectra were obtained using a Bruker Avance DRX 500 MHz (Germany) in deuterated DMSO-d6 solvent and TMS as an internal reference. The expected structure of some synthesized new compounds was also determined based on their mass measurement. The mass spectra were obtained by using the Shimadzu TQ8040 (Japan). Thin-layer chromatography of the starting materials and products was performed by using Merck chromatography sheets (Germany). The spot was visualized by exposing the dry plate to UV light

2.2 Synthesis Methods

2.2.1 General Procedure for the Synthesis of New Compounds [A1-A8]

Six mmol of 3,3'-(1,4-phenylene) bis(1-(4-aminophenyl) prop-2-en-1-one) [A] prepared as mentioned in the literature [12] was dissolved in ethanol (25 ml) to make solution (1). In another flask, 6 mmol of carboxylic acid was dissolved in 10 ml of ethanol to make a solution (2). Then, solution (2) was added to the solution (1) in a three-neck round-bottom flask with constant stirring. After 20 minutes, 20 ml of 10% aqueous sodium hydroxide solution were added dropwise to the mixture, and then the mixture was refluxed at 80-90 °C for about 6 hours in an oil bath with constant stirring [13]. The reactants and their amounts are listed in Table 1. The reaction was monitored by TLC using an (8:2) [methanol-benzene] eluent. After the reaction was completed, the reaction mixture was cooled to room temperature. The reaction mixture was poured into 150 ml of cold water and the precipitated solid was filtered off, washed several times with water until

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the filtrate was neutral to litmus, and dried. The obtained product powder had a different color and was recrystallized with a mixture of ethanol and water (1:1).

2.2.2 Synthesis of N-(4-(3-(4-(3-(4-aminophenyl)-3-oxoprop-1-enyl) phenyl) acryloyl) phenyl) palmitamide [A1]

This compound is produced by reaction compound [A] with a palmitic acid yield of 77.6% and a melting point of 172-174 °C as shown in Scheme 1. M/z [M^+] = 606, $C_{40}H_{50}N_2O_3$, FT-IRν (cm^{-1}) [14-16]: 1708 (C=O) amide and ketone, 3329-3429 (–NH_2), 3221 (–NH) amide, 1635 (C=C) aliphatic, 3113 (C-H) aromatic. δ 1H-NMR (DMSO-d6/ppm) [15-19]: 1.48-1.90 (m, 26H, −(CH)−), 2.94-2.98 (t, 3H, −CH_3), 3.73-3.79 (t, 2H, −CH_2), 4.03 (s, 2H, −NH_2), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH).

2.2.3 Synthesis of 12-oxo-12-(4-(3-(4-(3-oxo-3-(4-Hexadecanamidophenyl) prop-1-enyl) phenyl) acryloyl) phenylamino) dodecanoic acid [A2]

The reaction compound [A1] with dodecanedioic acid synthesizes this compound as represented in Scheme 2. After purification, a light orange powder was obtained. The product was obtained at a yield of 76% and a melting point of 190–193 °C. M/z [M+] = 819 $C_{52}H_{70}N_2O_6$, FT-IR ν (cm^{-1}) [14-16]: 1724 (C=O) amide and ketone, 3444 (–OH), 3425 (–NH) amide, 1624 (C=C) aliphatic, 3220 (C-H) aromatic. δ 1H-NMR (DMSO-d6 /ppm) [15, 17-19] 1.48-1.90 (m, 55H, −(CH)−), 2.94-2.98 (t, 3H, –CH_3), 3.73-3.79 (t, 2H, –CH_2), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH).

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Table 1: The prepared amide derivatives.

Derivative Code	Product Name	Reactant 1	Reactant 2
A1	N-(4-(3-(4-(3-(4-aminophenyl)-3-oxoprop-1-enyl) phenyl) acryloyl) phenyl) palmitamide	3,3'-(1,4-phenylene)bis(1-(4-aminophenyl) prop-2-en-1-one) [A] (6 m mole)	Palmitic acid (6 m mole)
A2	12-oxo-12-(4-(3-(4-(3-oxo-3-(4- Hexa decanamidophenyl) prop-1-enyl) phenyl) acryloyl) phenylamino) dodecanoic acid	A1 (6 mmole)	Dodecanedioic acid (6 mmole)
A3	10-oxo-10-(4-(3-(4-(3-oxo-3-(4-Hexa decanamidophenyl) prop-1-enyl) phenyl) acryloyl) phenylamino) decanoic acid	A1 (6 mmole)	Decanedioic acid (6 mmole)
A4	7-oxo-7-(4-(3-(4-(3-oxo-3-(4-Hexadecanamidophenyl) prop-1-enyl) phenyl) acryloyl) phenylamino) heptanoic acid	A1 (6 mmole)	Heptanedioic acid (6 mmole)
A5	N-(4-(3-(4-(3-(4-acrylamidophenyl)-3-oxo prop-1-enyl) phenyl) acryloyl) phenyl) hexadecanamide	A1 (6 mmole)	Acrylic acid (6 mmole)
A6	N^1-acryloyl-N^{12}-(4-(3-(4-(3-oxo-3-(4-palmitamido phenyl) prop-1-enyl)phenyl) acryloyl) phenyl) dodecane diamide	A2 (6 mmole)	Acrylamide (6 mmole)
A7	N^1-acryloyl-N^{10}-(4-(3-(4-(3-oxo-3-(4-palmitamido phenyl) prop-1-enyl)phenyl) acryloyl)phenyl) decane diamide	A3 (6 mmole)	Acrylamide (6 mmole)
A8	N^1-acryloyl-N^7-(4-(3-(4-(3-oxo-3-(4-palmitamido phenyl)prop-1-enyl)phenyl) acryloyl) phenyl) heptane diamide	A4 (6 mmole)	Acrylamide (6 mmole)

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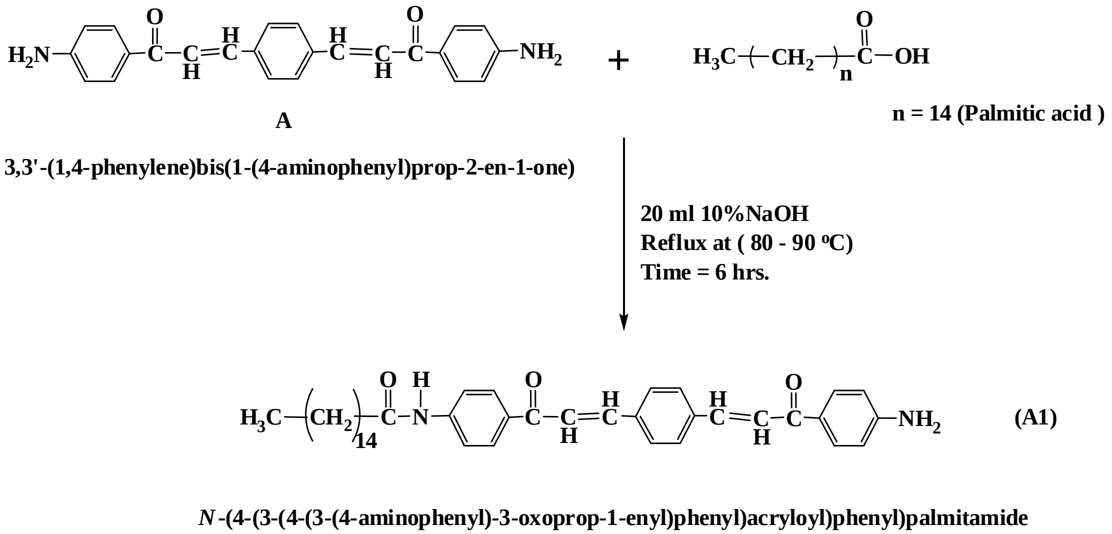
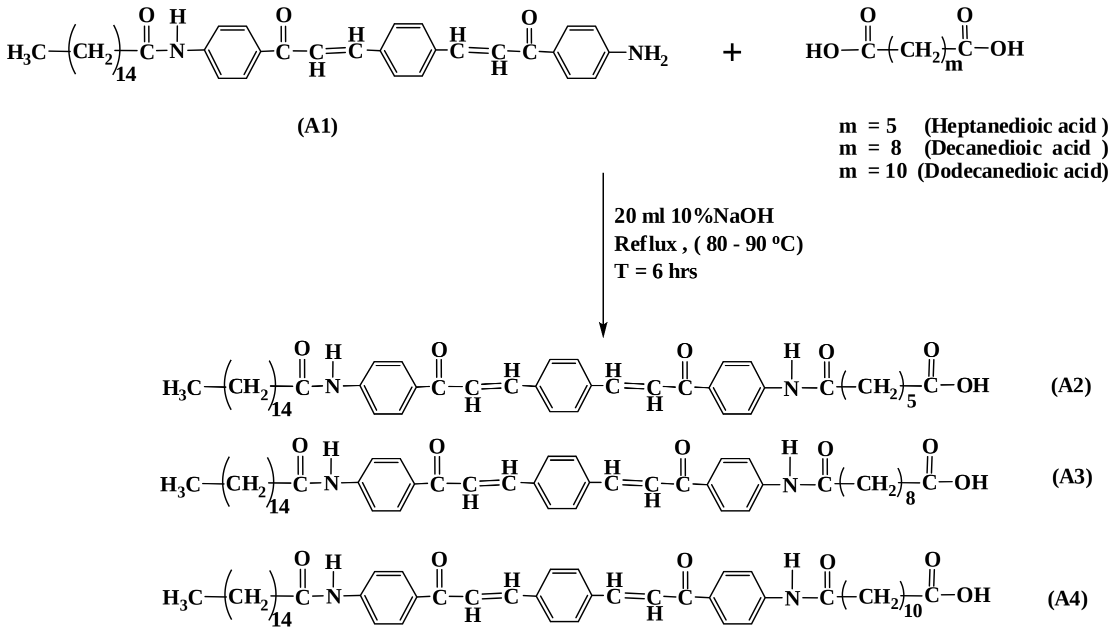


Scheme 1. Synthesis route of a derivative A1.



Scheme 2. Route of synthesis of derivatives A2, A3, and A4.

2.2.4 Synthesis of 10-oxo-10-(4-(3-(4-(3-oxo-3-(4-Hexadecanamidophenyl) prop-1-enyl) phenyl) acryloyl) phenylamino)decanoic acid [A3]

This compound was synthesized by the reaction of [A1] with decanedioic acid as shown in Scheme 2. After purification, a light orange powder was obtained at a yield of 72% and a melting point of 184-187 °C. M/z [M+] = 791 $C_{50}H_{66}N_2O_6$, FT-IR ν (cm^{-1}) [14-16]: 1724 (C=O) amide and ketone, 3442 (–OH), 3250 (–NH) amide, 1640 (C=C) aliphatic, 3220 (C-H) aromatic. δ 1H-NMR (DMSO-

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d6/ ppm) [15-19]: 1.48-1.90 (m, 55H, −(CH)−), 2.94-2.98 (t, 3H, $–CH_3$), 3.73-3.79 (t, 2H, $–CH_2$), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH).

2.2.5 Synthesis of 7-oxo-7-(4-(3-(4-(3-oxo-3-(4-hexadecanamidophenyl) prop-1-enyl) phenyl) acryloyl) phenylamino)heptanoic acid [A4]

This compound was prepared by reacting [A1] with heptanedioic acid, as shown in Scheme 2. After purification, a light orange powder was obtained at a yield of 73.3% and a melting point of 181–183 °C. M/z [M+] = 749 $C_{47}H_{60}N_2O_6$, FT-IR ν (cm^{-1}) [14-16]: 1724 (C=O) amide and ketone, 3425 (–OH), 3425 (–NH) amide, 1660 (C=C) aliphatic, 3210 (C-H) aromatic. δ 1H-NMR (DMSO-d6 /ppm) [15-19]: 1.48-1.90 (m, 55H, −(CH)−), 2.94 - 2.98 (t, 3H, $–CH_3$), 3.73 -3.79 (t, 2H, $–CH_2$), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH).

2.2.6 Synthesis of N-(4-(3-(4-(3-(4-acrylamidophenyl)-3-oxoprop-1-enyl) phenyl) acryloyl) phenyl) hexadecanamide [A5]

This compound was prepared by reacting [A1] with acrylic acid, as shown in Scheme 3. A light yellow powder was obtained with a yield of 77% and a melting point of 184–186 °C after purification. M/z [M+] = 660 $C_{43}H_{52}N_2O_4$, FT-IR ν (cm^{-1}) [14-16]: 1724 (C=O) amide and ketone, 3425 (–OH), 3425 (–NH) amide, 1660 (C=C) aliphatic, 3210 (C-H) aromatic. δ 1H-NMR (DMSO-d6 /ppm) [15, 17, 18, 19]: 1.48-1.90 (m, 55H, −(CH)−), 2.94 - 2.98 (t, 3H, $–CH_3$), 3.73 -3.79 (t, 2H, $–CH_2$), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH).

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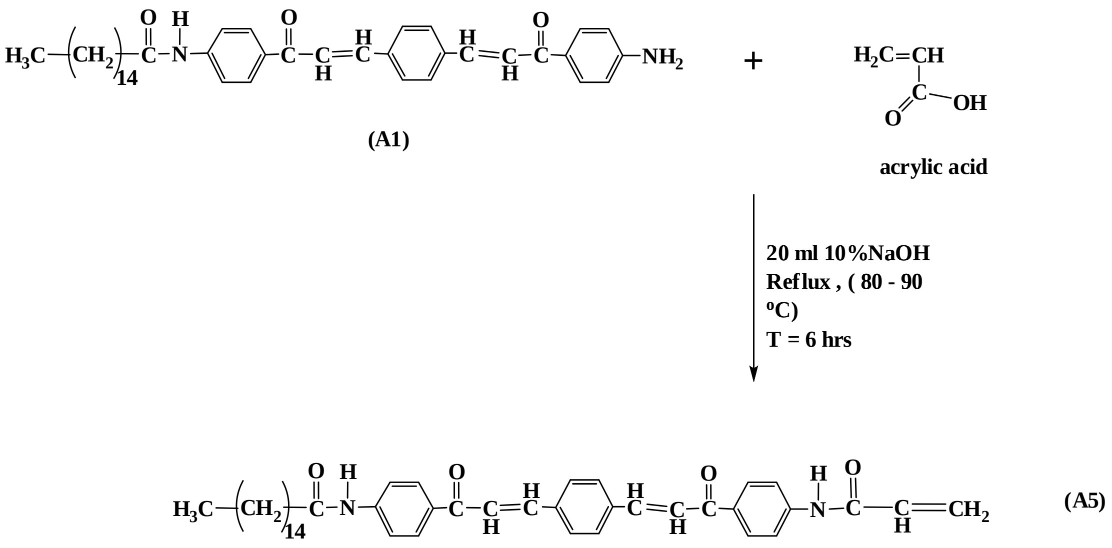

Scheme 3: Synthesis route of a derivative A5.

2.2.7 Synthesis of N^1-acryloyl-N^{12}-(4-(3-(4-(3-oxo-3-(4-palmitamido phenyl) prop-1-enyl) phenyl) acryloyl) phenyl) dodecane diamide [A6]

This compound was prepared by reacting [A2] with acrylamide, as shown in Scheme 4. After purification, a light yellow powder was obtained at a yield of 73 % and a melting point of 199-203 °C. M/z [M+] = 872 $C_{55}H_{73}N_3O_6$, FT-IR ν (cm^{-1}) [14-16]: 1724 (C=O) amide and ketone, 3425 (–OH), 3425 (–NH) amide, 1660 (C=C) aliphatic, 3210 (C-H) aromatic. δ 1H-NMR (DMSO-d6 /ppm) [15-19]: 1.48-1.90 (m, 55H, −(CH)−), 2.94 - 2.98 (t, 3H, $–CH_3$), 3.73 -3.79 (t, 2H, $–CH_2$), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH), 13 (s, 1H, -NH).

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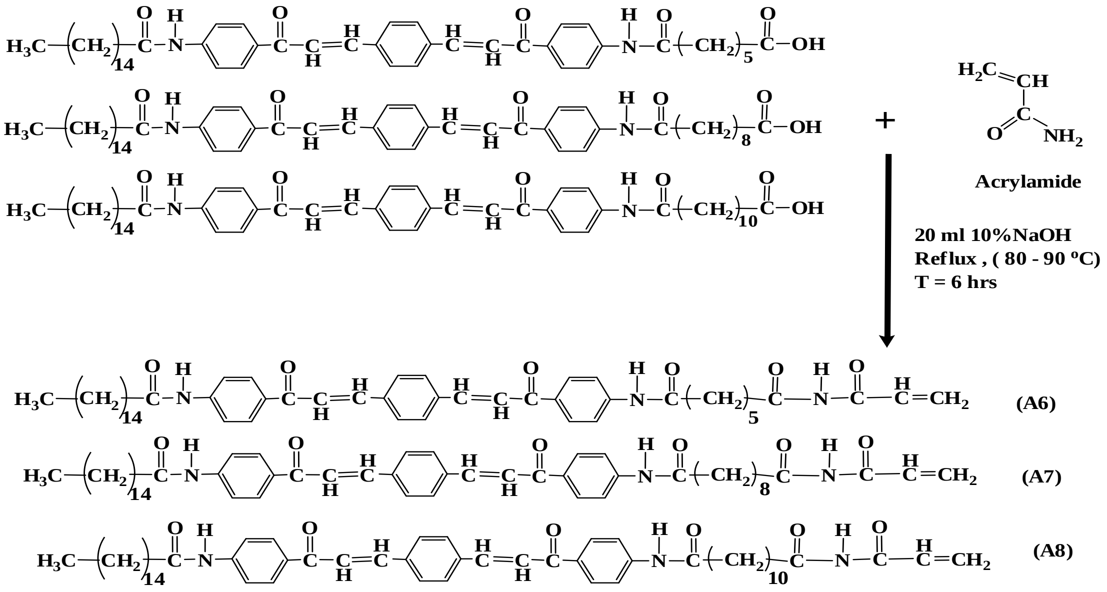

Scheme 4. Route of synthesis of derivatives A2, A3, and A4.

2.2.8 Synthesis of N^1-acryloyl-N^{10}-(4-(3-(4-(3-oxo-3-(4-palmitamido phenyl) prop-1-enyl)phenyl) acryloyl) phenyl) decane diamide [A7]

This compound is synthesized by reacting [A3] with acrylamide as shown in Scheme 4. A light yellow powder was obtained at a yield of 71 % and a melting point of 198-200 °C after purification. M/z [M+] = 844 $C_{53}H_{69}N_3O_6$, FT-IR ν (cm^{-1}) [14-16]: 1724 (C=O) amide and ketone, 3425 (–OH), 3425 (–NH) amide, 1660 (C=C) aliphatic, 3210 (C-H) aromatic. δ 1H-NMR (DMSO-d6 /ppm) [15-19]: 1.48-1.90 (m, 55H, −(CH)−), 2.94 - 2.98 (t, 3H, –CH_3), 3.73 -3.79 (t, 2H, –CH_2), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH), 13 (s, 1H, -NH.

2.2.9 Synthesis of N^1-acryloyl-N^7-(4-(3-(4-(3-oxo-3-(4-palmitamido phenyl) prop-1-enyl)phenyl) acryloyl) phenyl) heptane diamide [A8]

This compound was prepared by reacting [A4] with acrylamide, as represented in Scheme 4. After purification, a light yellow powder was obtained with a yield of 74 % and 195-197 °C melting point. M/z [M+] = 802 $C_{50}H_{63}N_3O_6$, ν (cm^{-1}) [14-16]: 1724 (C=O) amide and ketone, 3425 (–OH), 3425 (–NH) amide, 1660 (C=C) aliphatic, 3210 (C-H) aromatic. δ ^{1}HNMR (DMSO-d6/ppm) [15-19]: 1.48-1.90 (m, 55H, −(CH)−), 2.94-2.98 (t, 3H, –CH_3), 3.73 -3.79 (t, 2H, –CH_2), 5.77-5.97

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(dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH), 13 (s, 1H, -NH).

3. Results and Discussion

The eight-amide derivatives [A1-A8] were prepared by direct condensation of reactant 1 and reactant 2, as shown in Table (1). They were characterized first by FTIR spectroscopy. The reaction was followed by the appearance of the absorption bands in the range of (1708–1724) cm^{-1} due to the presence of (C=O) stretching. The appearance of peaks in the range (2850–2920) cm^{-1} is attributed to (C-H) aliphatic stretching, and absorption bands in the range (1670–1624) cm^{-1} are assigned to (C=C) aliphatic stretching. While the absorption band of aromatic (C-H) stretching appears within the range of (3113–3260) cm^{-1}, the band within the range of (1427–1630) cm^{-1} is assigned to (C=C) aromatic stretching. The absorption bands within the range of (3217–3444) cm^{-1} due to the (-NH) stretching of the amide group and the bending (-NH) are shown within the range of (1543–1566) cm^{-1}. FTIR spectra also showed absorption bands at (3329, 3429) cm^{-1} due to symmetric and asymmetric (–NH2) stretching in the [A1] compound. The compounds [A2, A3, and A4] demonstrated the disappearance of stretching bands of (NH2) that appeared in (3329, 3429) cm^{-1} and the appearance of new bands due to (O-H) stretching at (3421-3444) cm^{-1}. The compound [A5] demonstrates the disappearance of stretching bands for (NH2) that appeared in (3329, 3429) cm^{-1}, as well as the appearance of a new band for stretching bands (NH amide and imide group) at (3425) cm^{-1}. As shown in Figures 1–8, [A6, A7, and A8] demonstrated the disappearance of stretching bands for (OH) that appeared in (3421-3444) cm^{-1} and the appearance of new stretching bands (NH, amide, and imide group) at (3421) cm^{-1}.

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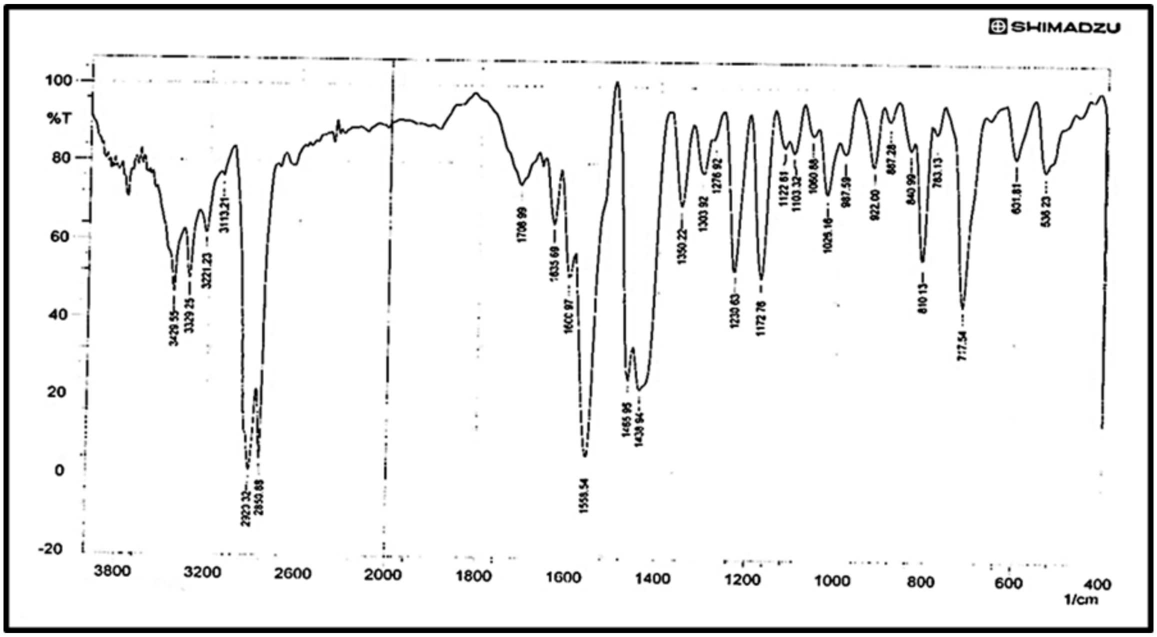

Figure 1: FT-IR of amide derivative A1.

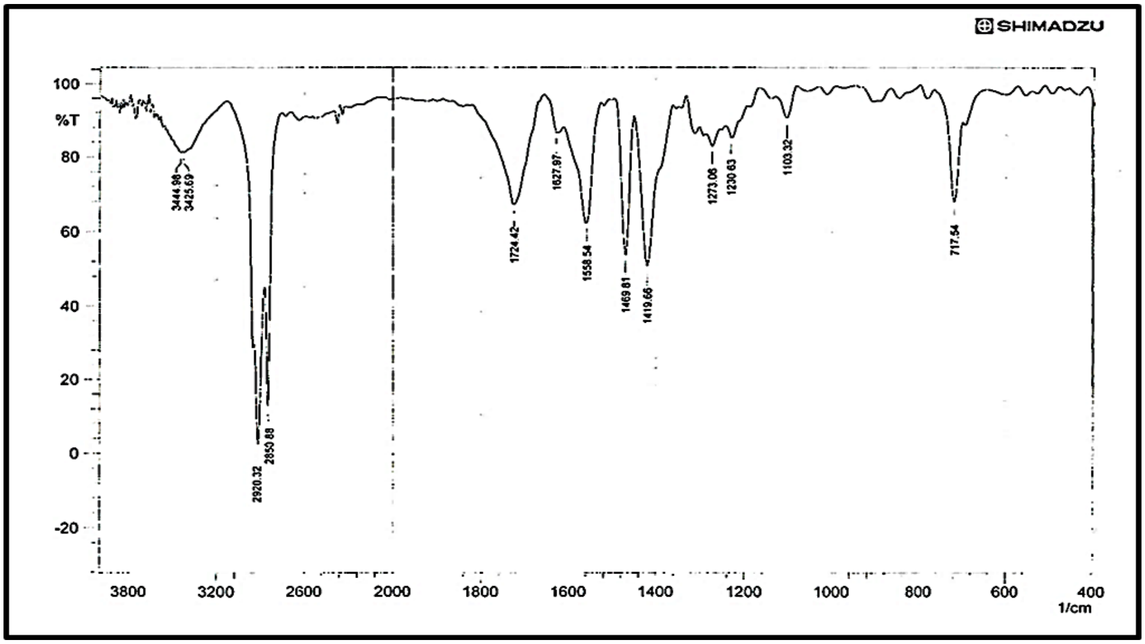

Figure 2: FT-IR of amide derivative A2.

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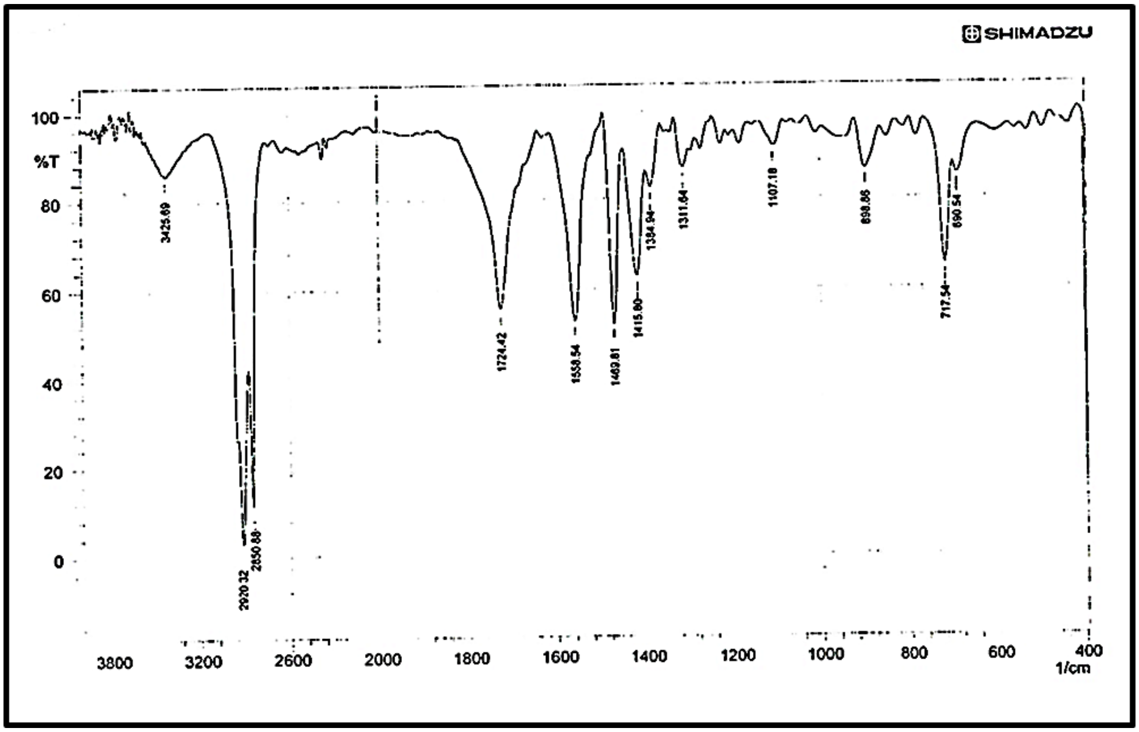

Figure 3: FT-IR of amide derivative A3.

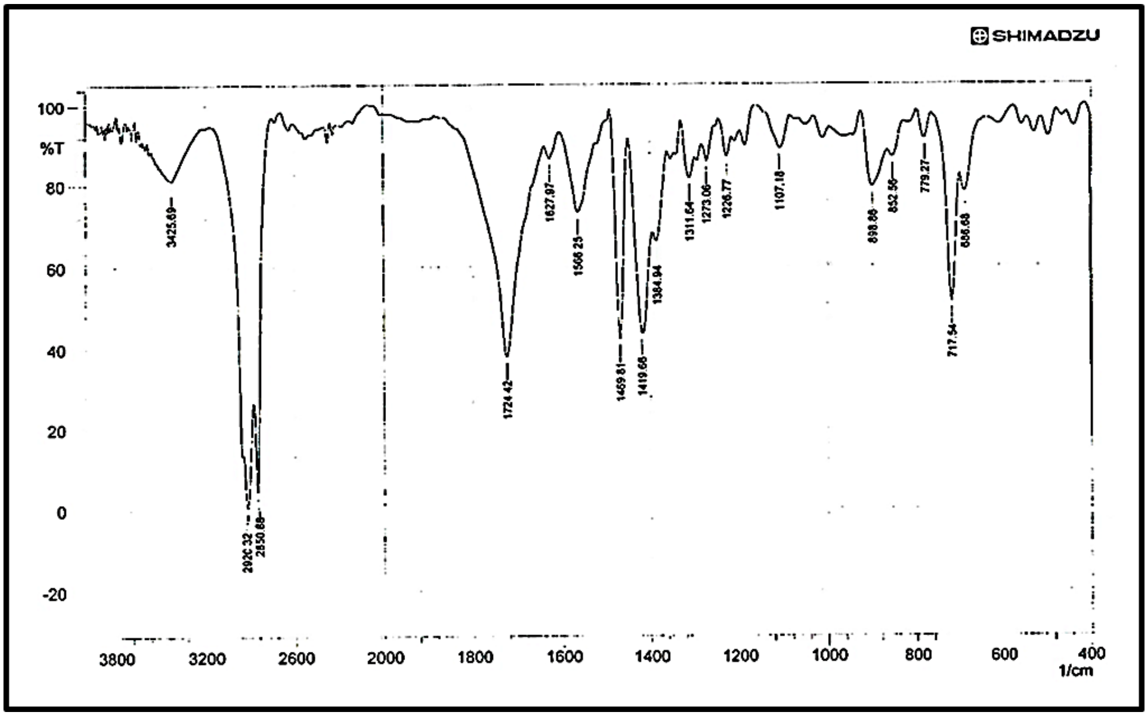

Figure 4: FT-IR of amide derivative A4.

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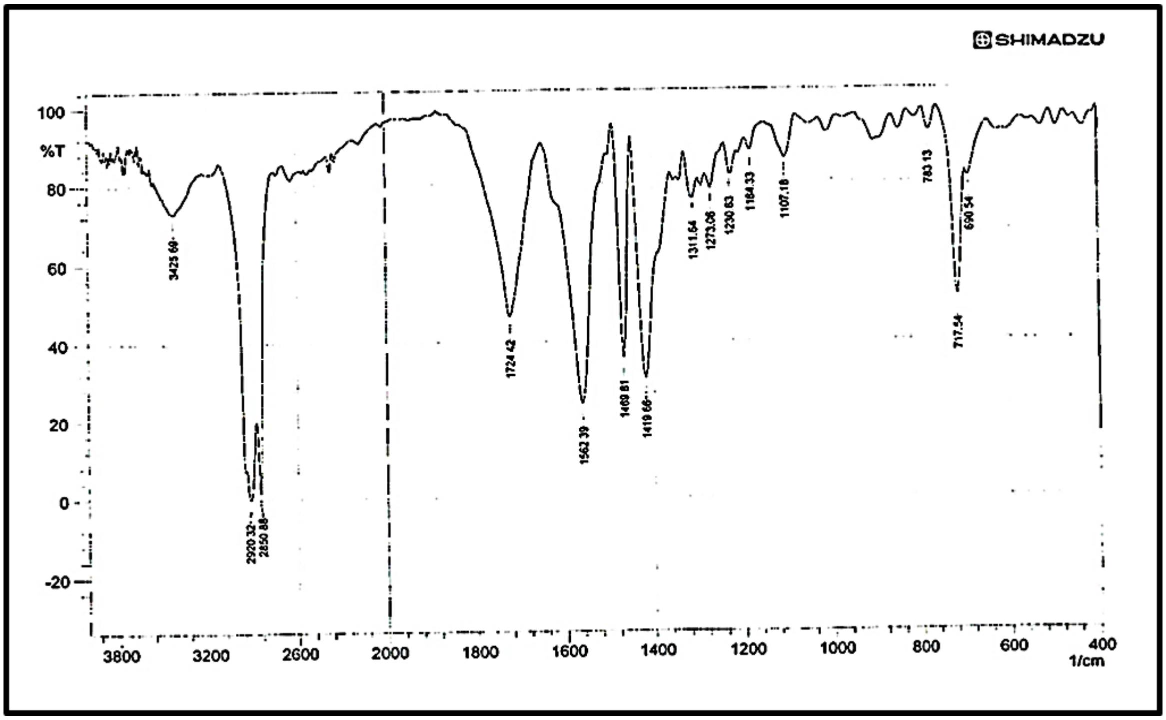

Figure 5: FT-IR of A5 amide derivative.

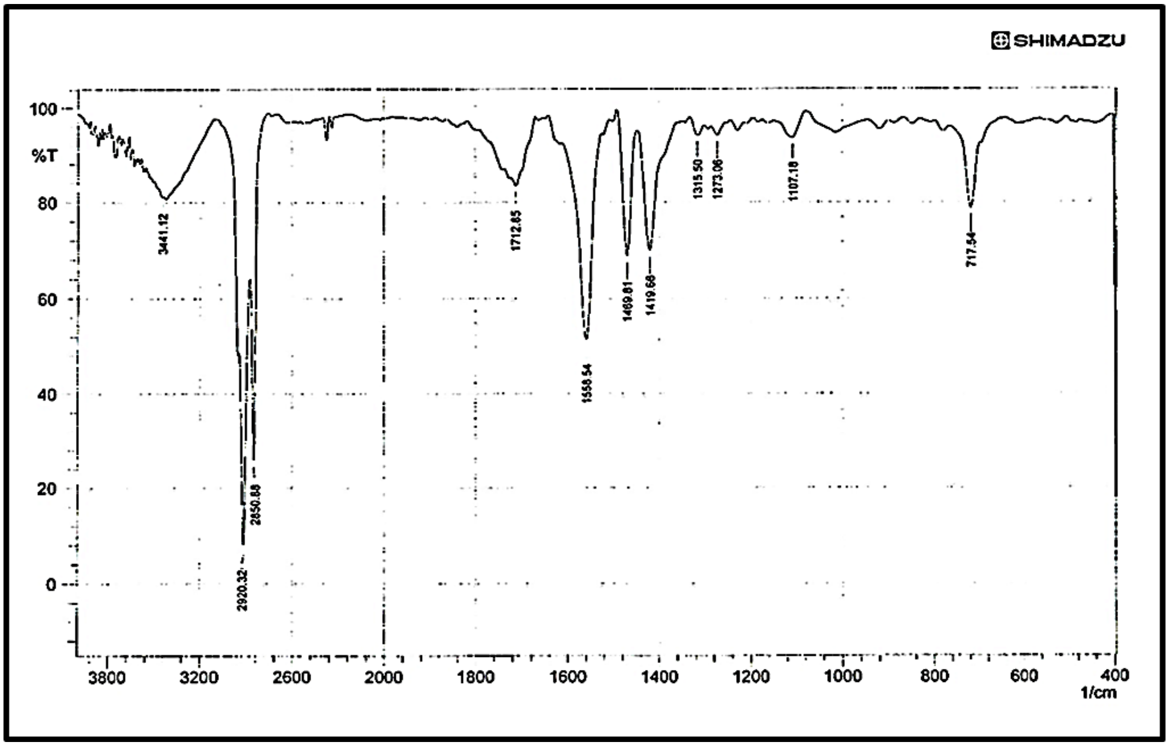

Figure 6: FT-IR of A6 amide derivative.

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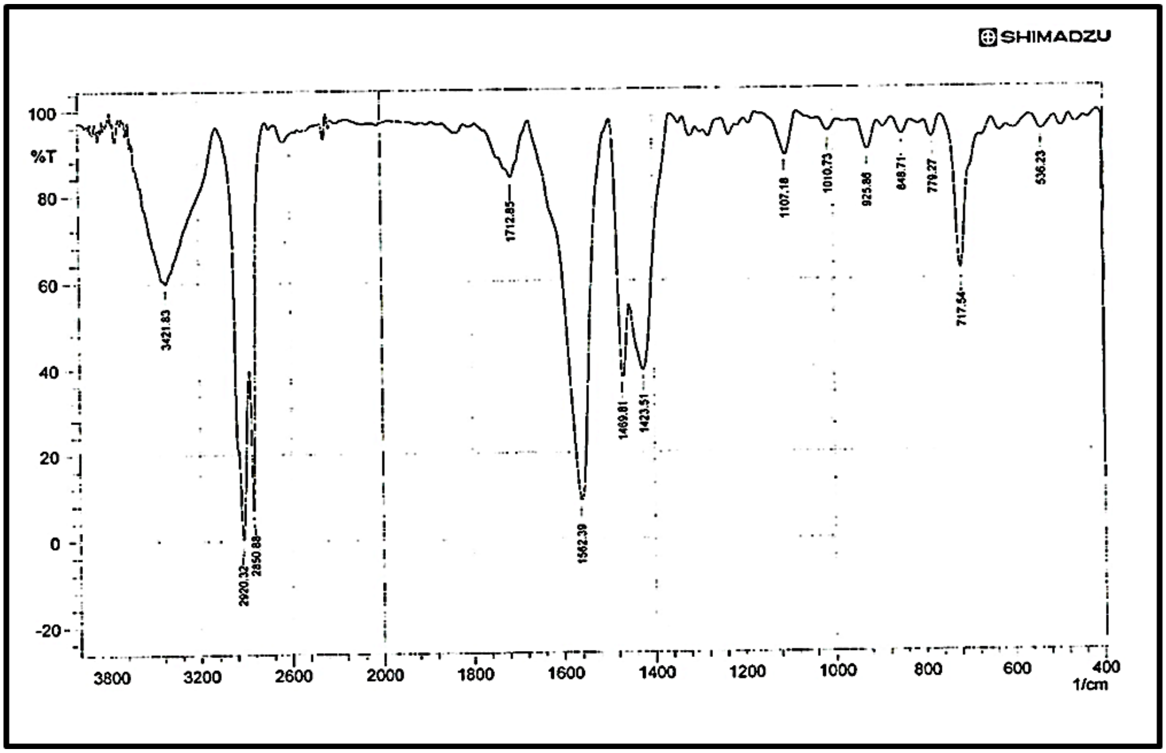

Figure 7: FT-IR of amide derivative A7.

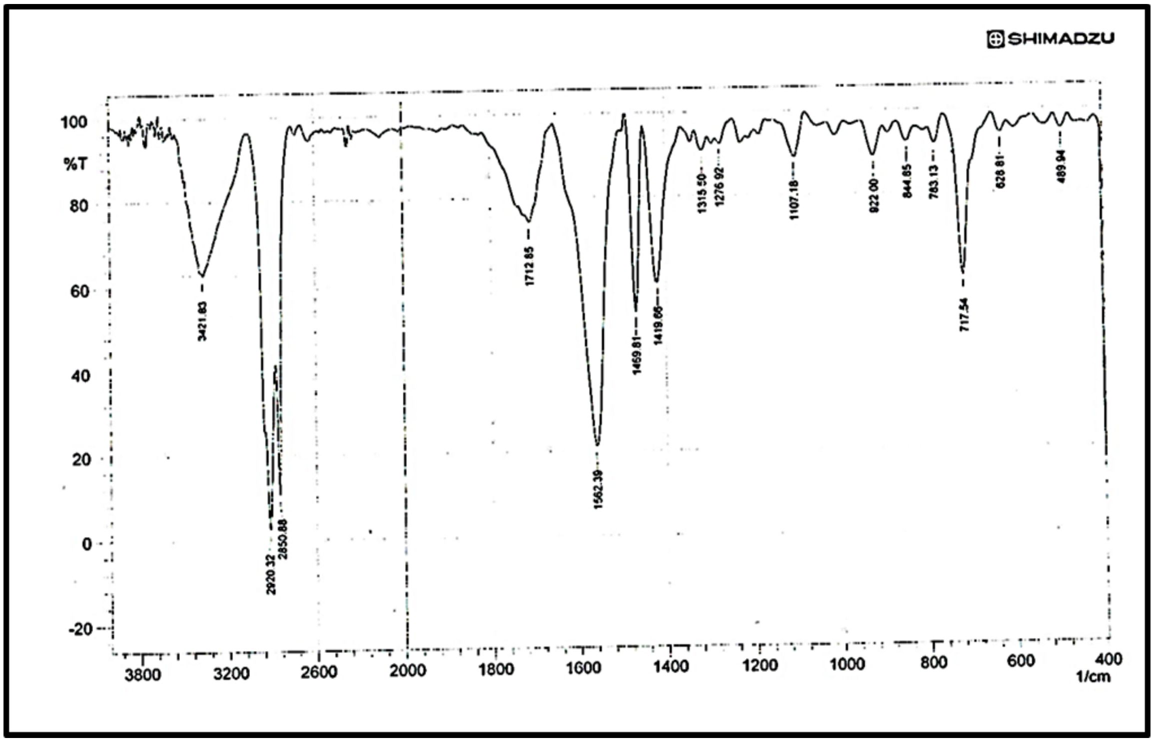

Figure 8: FT-IR of amide derivative A8.

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The 1H-NMR spectra of all the synthesized compounds were measured in deuterated dimethyl sulfoxide (DMSO-d6), and their chemical structures and their proton assignments are shown in Table (2). All spectra showed peaks in the region of (2.51) ppm, which were due to the DMSO solvent. All compounds [A1-A8] exhibited a singlet signal at (10.00) ppm due to a single proton (2H and 19H) for the amide group. In the compounds (A2, A3, and A4), singlet signals at a region of 12.00 ppm were assigned due to one proton (20H) for the (OH) group of carboxylic acid. While in compounds, (A6, A7, and A8) showed a singlet signal in the region of 13.00 ppm due to one proton (24H) for the (NH) imide group. Due to the mutual attraction between these four protons (9aH, 10aH, 11aH, and 12aH), the H-NMR spectral showed a singlet signal for four protons for aromatic rings (a) within the region of 7.9 ppm. The four protons for the aromatic ring (b) in positions (3bH, 4bH, 5bH, and 6bH) showed doublet signals within the range (7.57–7.67) ppm due to the mutual attraction between two protons being (3bH) and (4bH) within the region (7.57–7.59) ppm. This is also true for two protons (5bH) and (6bH) within the region (7.65–7.67) ppm. Also within the range (6.00-6.75) ppm, the four protons for the aromatic ring (c) in positions (15cH, 16cH, 17cH, and 18cH) showed doublet signals due to mutual attraction between two protons (15cH) with (16cH) within the region (6.00-6.60) ppm, and mutual attraction between two protons (17cH) with (18cH) within the region (6.73-6.75) ppm. The 1H-NMR spectral showed doublet signals within the range of (5.77-5.97) ppm for the two protons of the double bond, (7H and 8H), which is due to the mutual attraction between these two protons. It also showed doublet signals within the range of (5.77–5.80) ppm for the two protons of the double bond, (13H and 14H), which is due to the mutual attraction between these two protons. The 1H-NMR spectrum of compound [A1] showed a singlet signal within the region of (4.03) ppm due to two protons (1H and 2H) for the (–NH_2) group. The four protons for the aromatic ring (b) in positions (3bH, 4bH, 5bH, and 6bH) showed doublet signals within the range (7.57–7.67) ppm due to the mutual attraction between two protons being (3bH) and (4bH) within the region (7.57–7.59) ppm. This is also true for two protons (5bH) and (6bH) within the region (7.65–7.67) ppm. Also within the range (6.00-6.75) ppm, the four protons for the aromatic ring (c) in positions (15cH, 16cH, 17cH, and 18cH) showed doublet signals due to mutual attraction between two protons (15cH) with (16cH) within the region (6.00-6.60) ppm, and mutual attraction between two protons (17cH) with (18cH) within the region (6.73-6.75) ppm.

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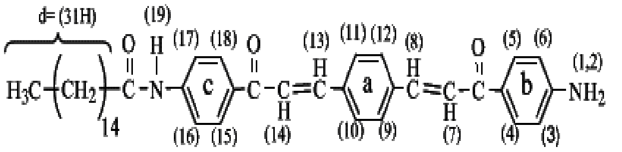
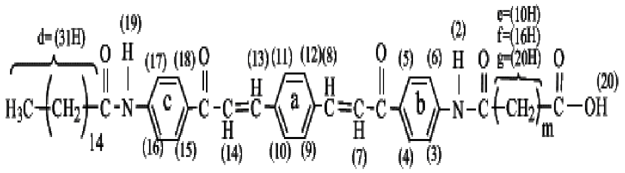
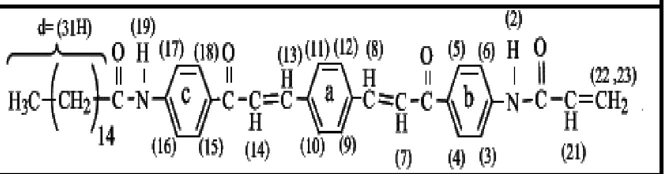
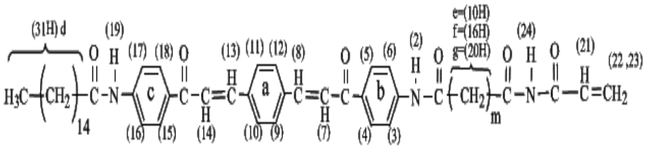
Table 2: Chemical structure and assigning of their protons.

Code	Product Name	Chemical Structure
A1	N-(4-(3-(4-(3-(4-aminophenyl)-3-oxoprop-1-enyl) phenyl) acryloyl) phenyl) palmitamide	
A2	12-oxo-12-(4-(3-(4-(3-oxo-3-(4-Hexa decanamidophenyl) prop-1-enyl) phenyl) acryloyl) phenylamino) dodecanoic acid	 m = 5 (Heptanedioic acid) m = 8 (Decanedioic acid) m = 10 (Dodecanedioic acid)
A3	10-oxo-10-(4-(3-(4-(3-oxo-3-(4-Hexa decanamidophenyl) prop-1-enyl) phenyl) acryloyl) phenylamino) decanoic acid	
A4	7-oxo-7-(4-(3-(4-(3-oxo-3-(4-Hexad ecanamidophenyl) prop-1-enyl) phenyl) acryloyl) phenylamino) heptanoic acid	
A5	N-(4-(3-(4-(3-(4-acrylamidophenyl)-3-oxo prop-1-enyl) phenyl) acryloyl) phenyl) hexadecanamide	
A6	N^1-acryloyl-N^{12}-(4-(3-(4-(3-oxo-3-(4-palmitamido phenyl)prop-1-enyl)phenyl) acryloyl) phenyl) dodecane diamide	 m= 5, 8, 10
A7	N^1-acryloyl-N^{10}-(4-(3-(4-(3-oxo-3-(4-palmitamido phenyl) prop-1-enyl)phenyl) acryloyl) phenyl) decane diamide	
A8	of N^1-acryloyl-N^7-(4-(3-(4-(3-oxo-3-(4-palmitamido phenyl) prop-1-enyl) phenyl) acryloyl) phenyl) heptane diamide	

The 1H-NMR spectral showed doublet signals within the range of (5.77-5.97) ppm for the two protons of the double bond, (7H and 8H), which is due to the mutual attraction between these two protons. It also showed doublet signals within the range of (5.77–5.80) ppm for the two protons of the double bond, (13H and 14H), which is due to the mutual attraction between these two protons. The 1H-NMR spectrum of compound [A1] showed a singlet signal within the region of (4.03) ppm due to two protons (1H and 2H) for the (–NH_2) group. The aliphatic protons showed multiple signals within the range of 1.48–1.90 ppm due to protons (dHs, eHs, fHs, and gHs). While compounds (A2, A3, and A4) showed triplet signals in the range (3.73–3.79) ppm due to (1dH, 2dH, 1eH, 2eH, 9eH, 10eH, 1fH, 2fH, 15fH, 16fH, 1gH, 2gH, and 20gH), compounds (A2, A3,

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and A4) showed triplet signals in the range (2.94–2.98) ppm due to three protons (29dH, 30dH, and 31dH). In the compounds (A5-A8), the proton (21 H) showed a triple signal within the range of (5.13-5.19) ppm due to the mutual attraction effect of the proton (21H) with the other two protons (22H) and (23H). While the two protons (22H and 23H) produced a double signal in the (4.47–4.50) ppm range due to the attraction effect of both protons (22H and 23H) with the proton (21H) as shown in Figures 9 -16.

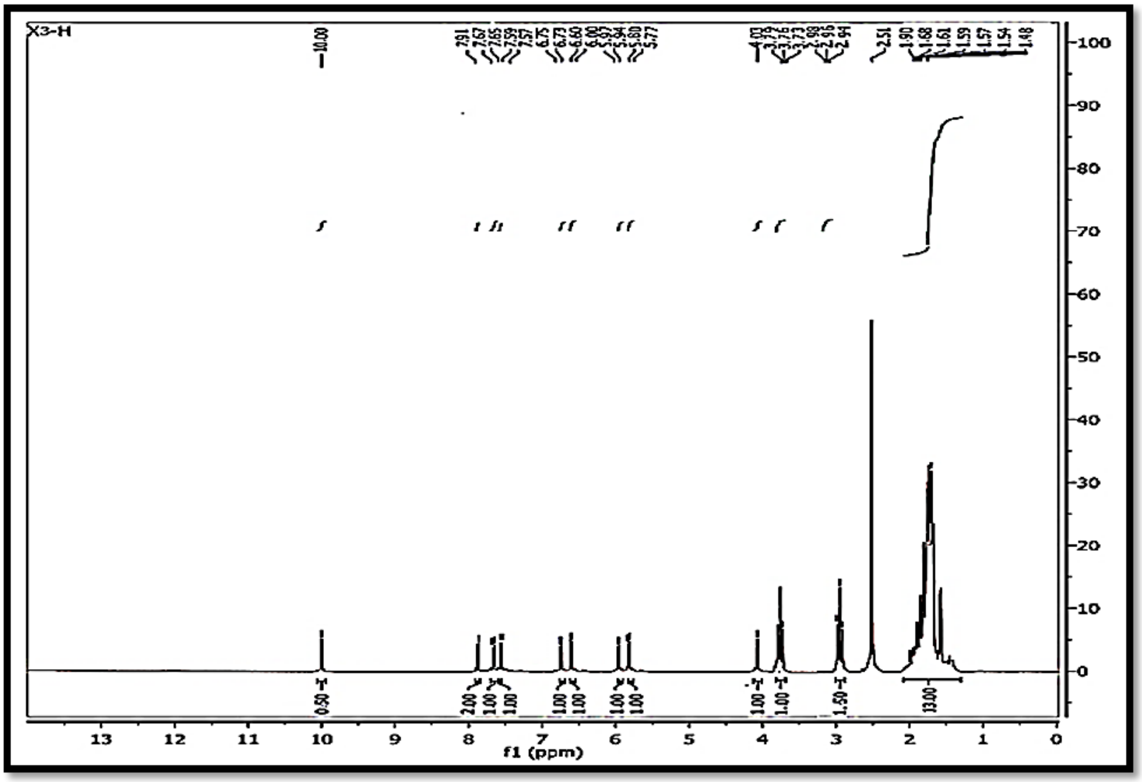

Figure 9: 1H-NMR spectrum of amide derivative A1.

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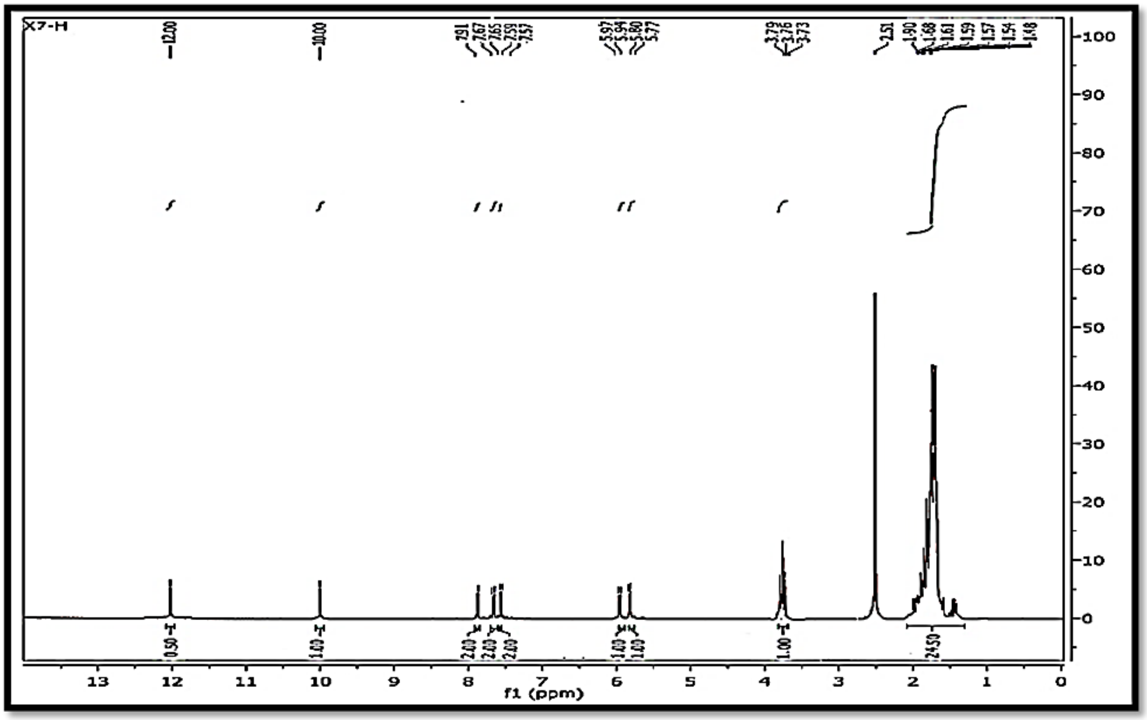

Figure 10: The 1H-NMR spectrum of amide derivative A2.

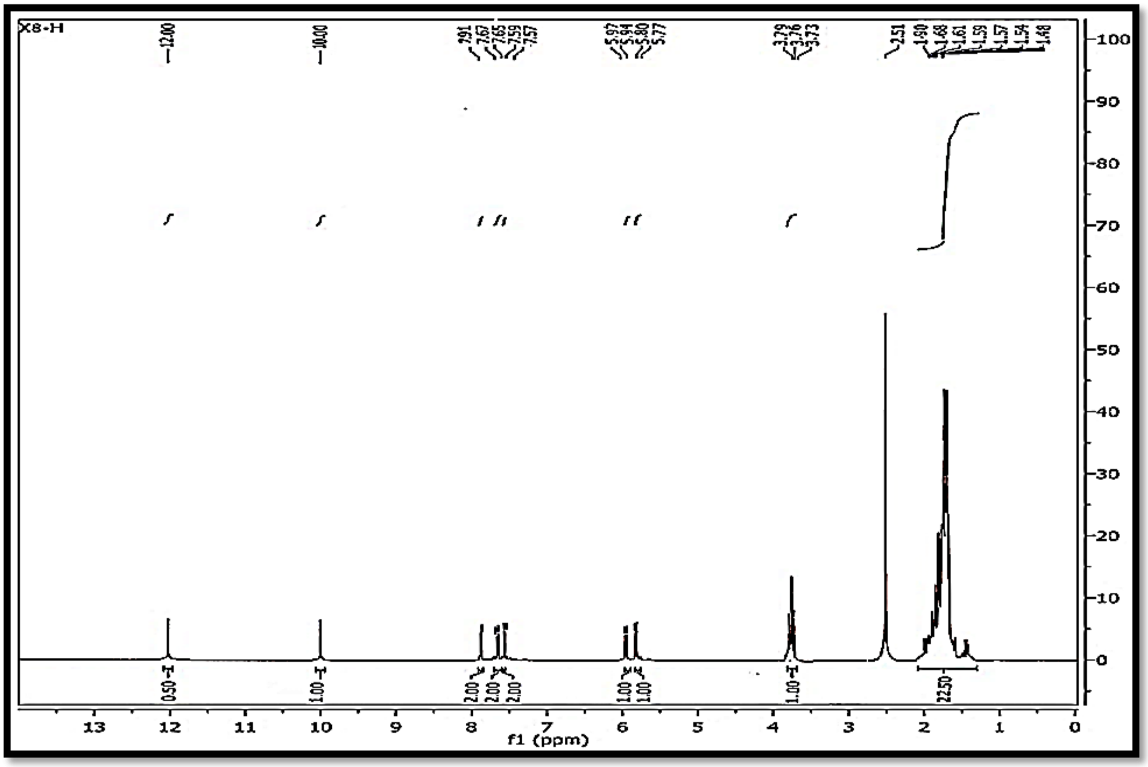

Figure 11: The 1H-NMR spectrum of amide derivative A3.

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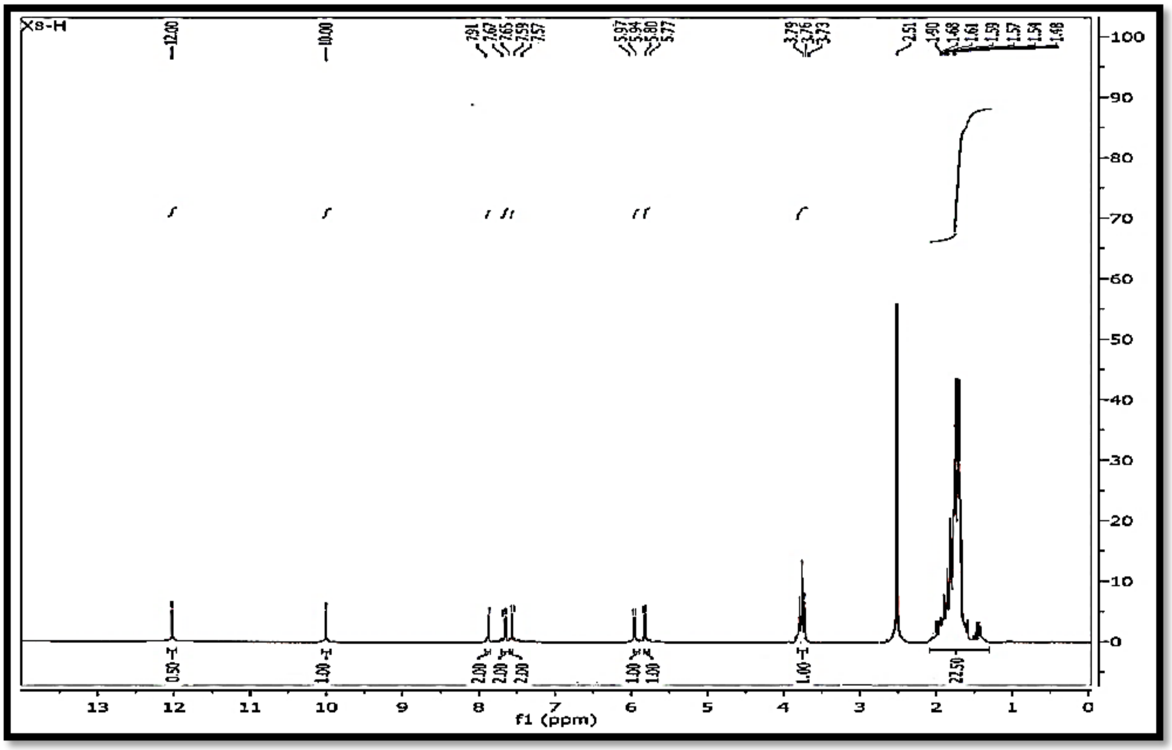

Figure 12: The 1H-NMR spectrum of amide derivative A4.

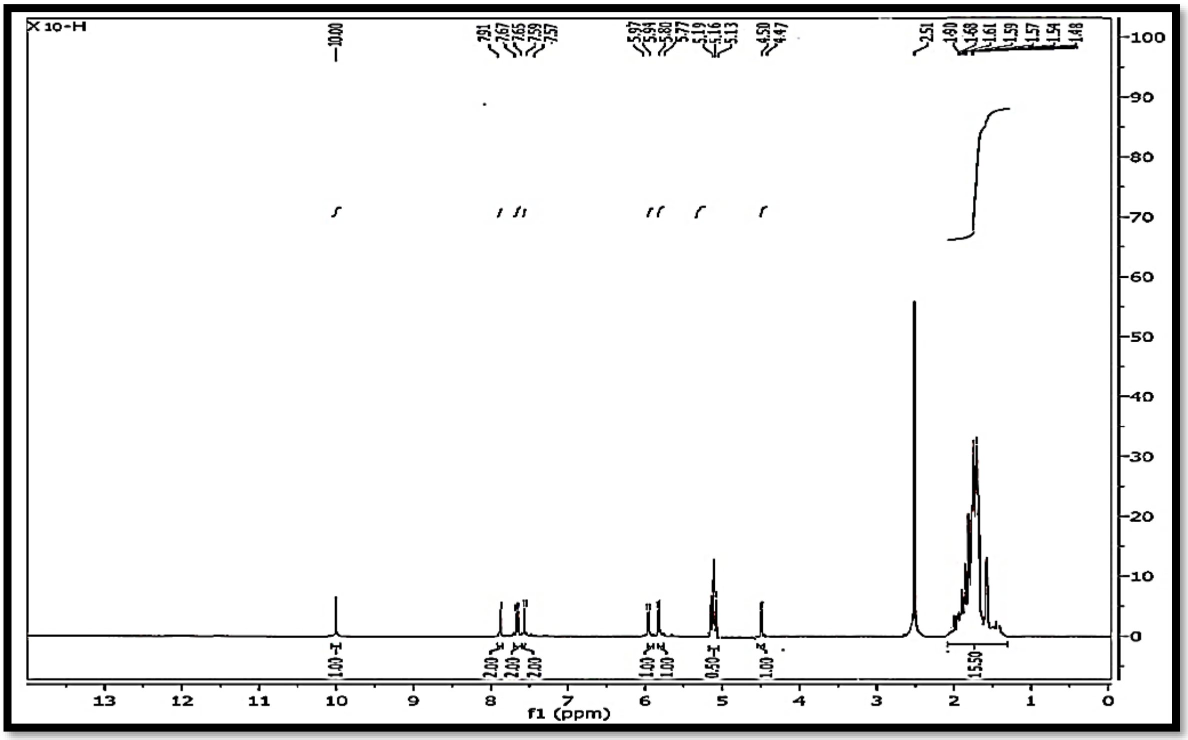

Figure 13: The 1H-NMR spectrum of amide derivative A5.

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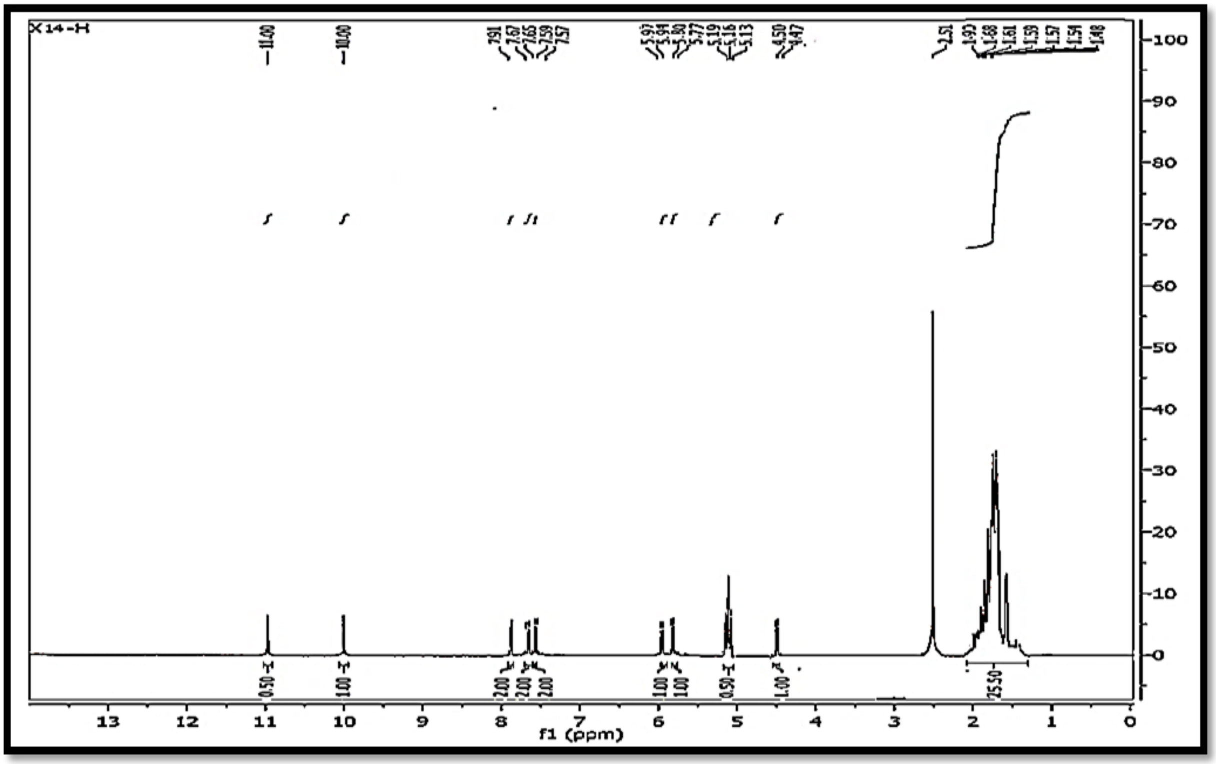

Figure 14: The 1H-NMR spectrum of amide derivative A6.

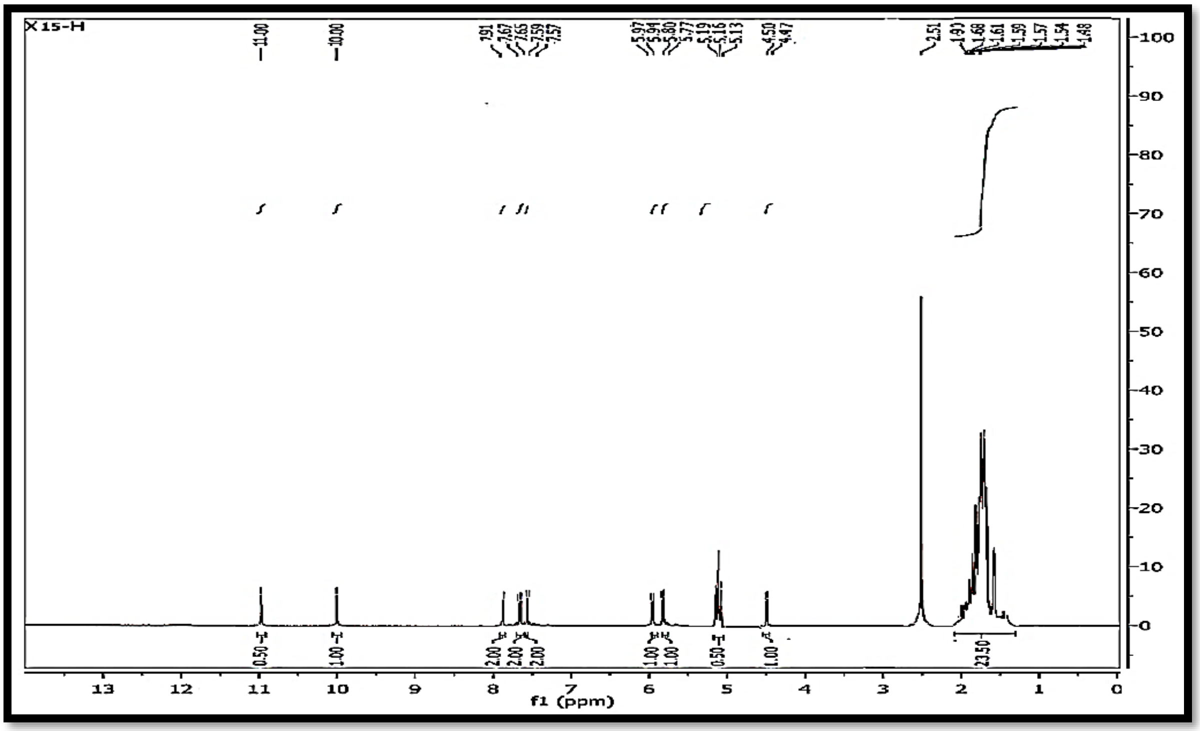

Figure 15: The 1H-NMR spectrum of amide derivative A7.

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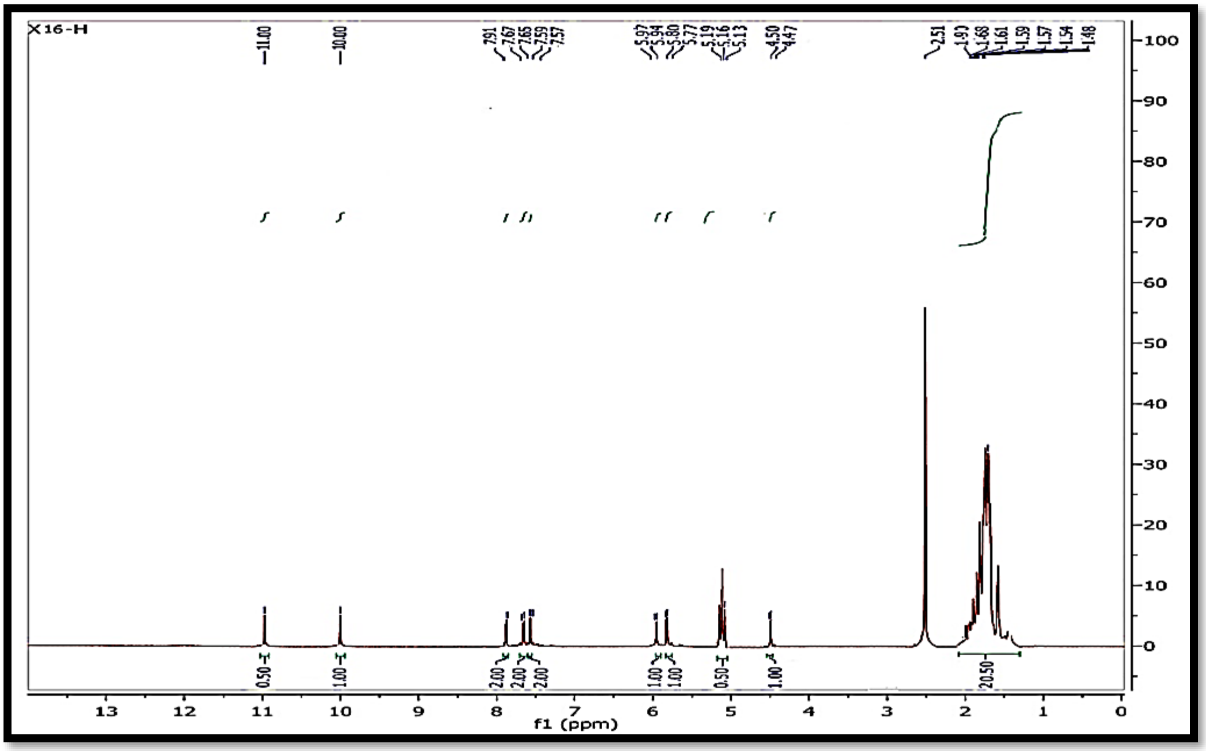

Figure 16: The 1H-NMR spectrum of amide derivative A8.

The mass spectra of the synthesized new compounds [A1-A8] are demonstrated in Figures 17-24. From the mass spectra, it was observed that the peak at (m/z = 606, 819, 790, 749, 660, 872, 844, and 802) represented the molecular ions [M+] for (AH1, AH2, AH3, AH4, AH5, AH6, AH7, and AH8) compounds, respectively. These peaks indicated that the structures of the synthesized compounds in this study were in agreement with our expectations.

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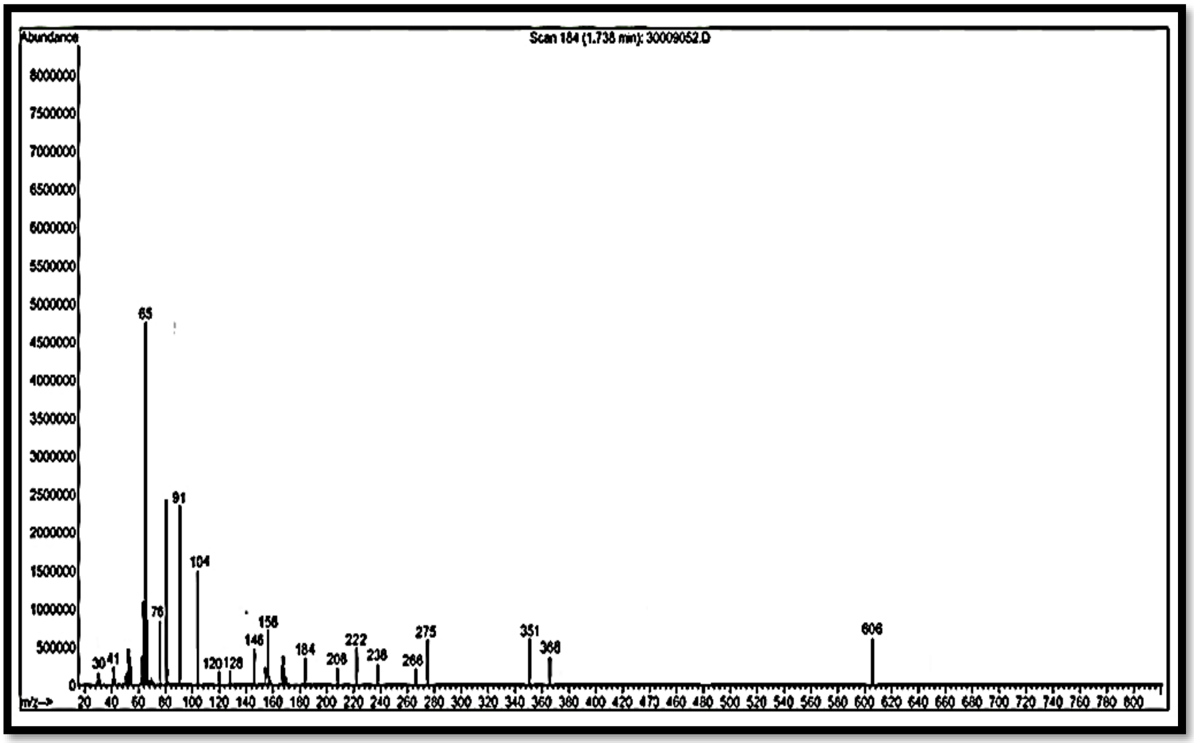

Figure 17: Mass spectrometry of amide derivative A1.

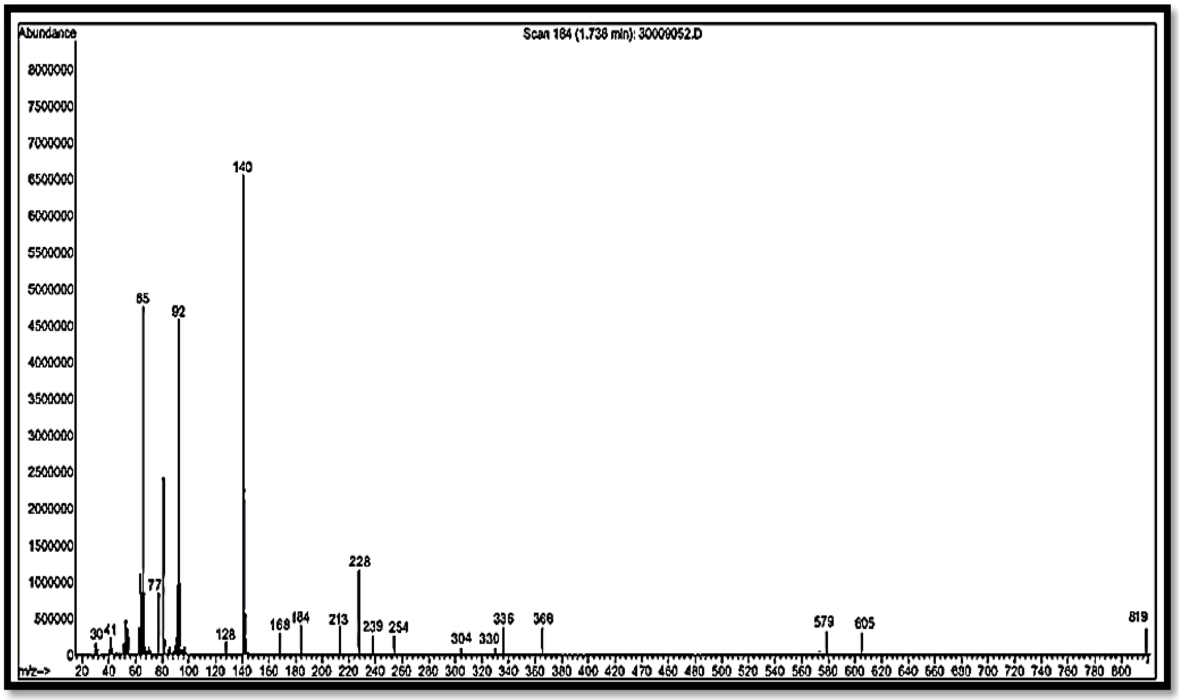

Figure 18: Mass spectrometry of amide derivative A2.

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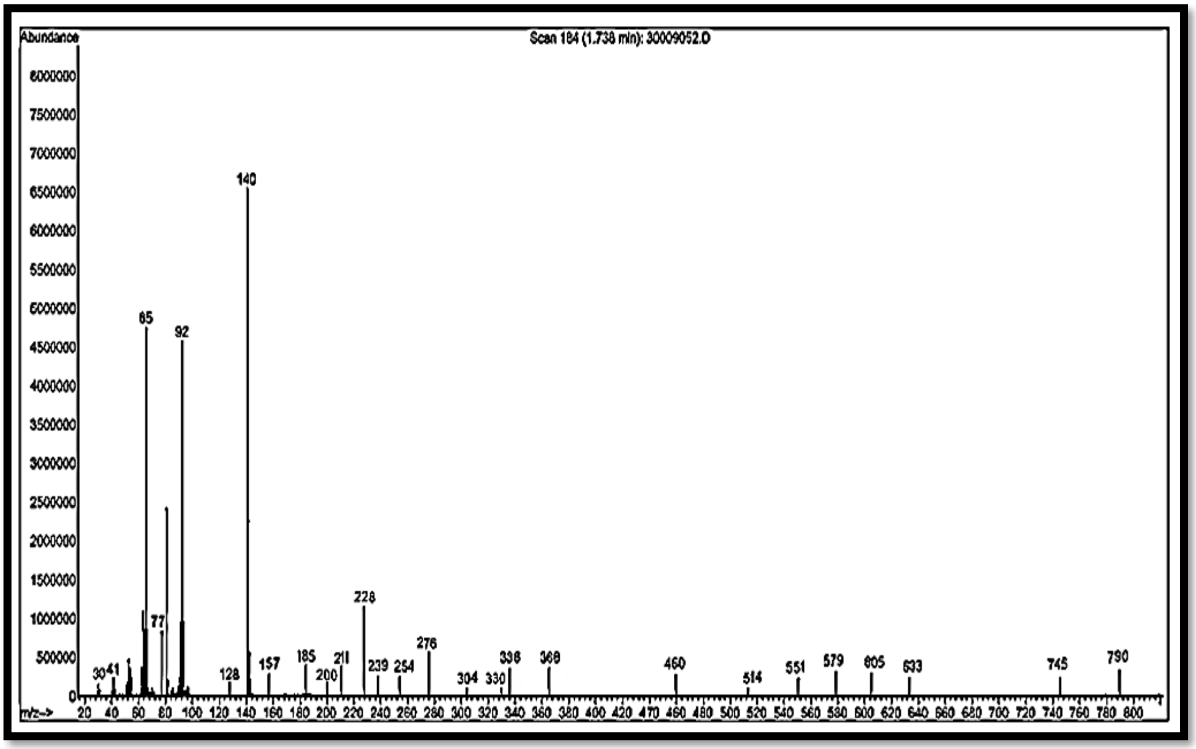

Figure 19: Mass spectrometry of amide derivative A3.

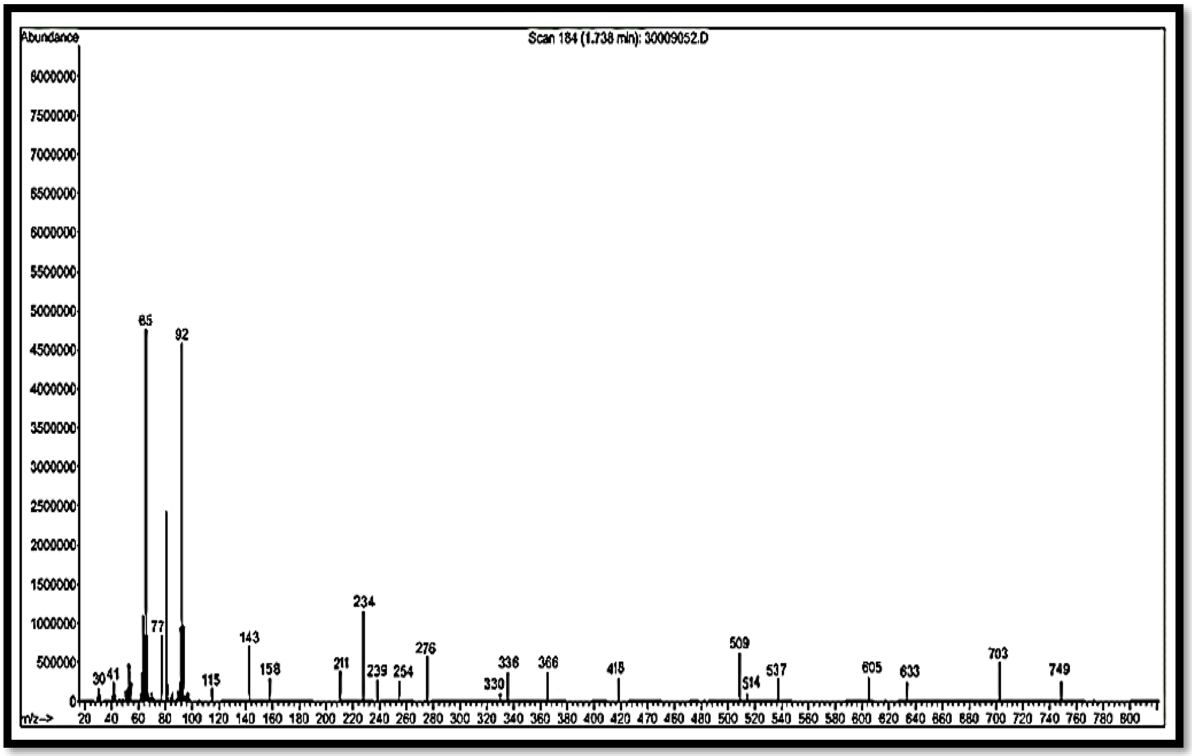

Figure 20: Mass spectrometry of amide derivative A4.

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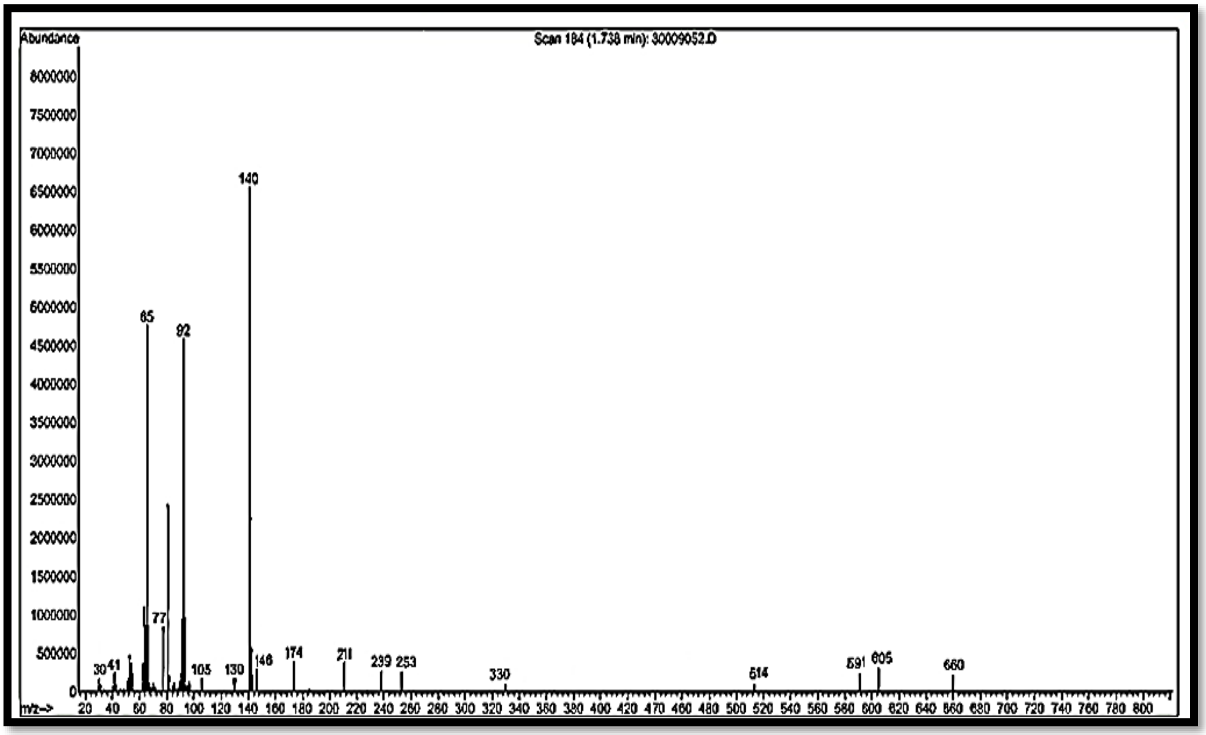

Figure 21: Mass spectrometry of amide derivative A5.

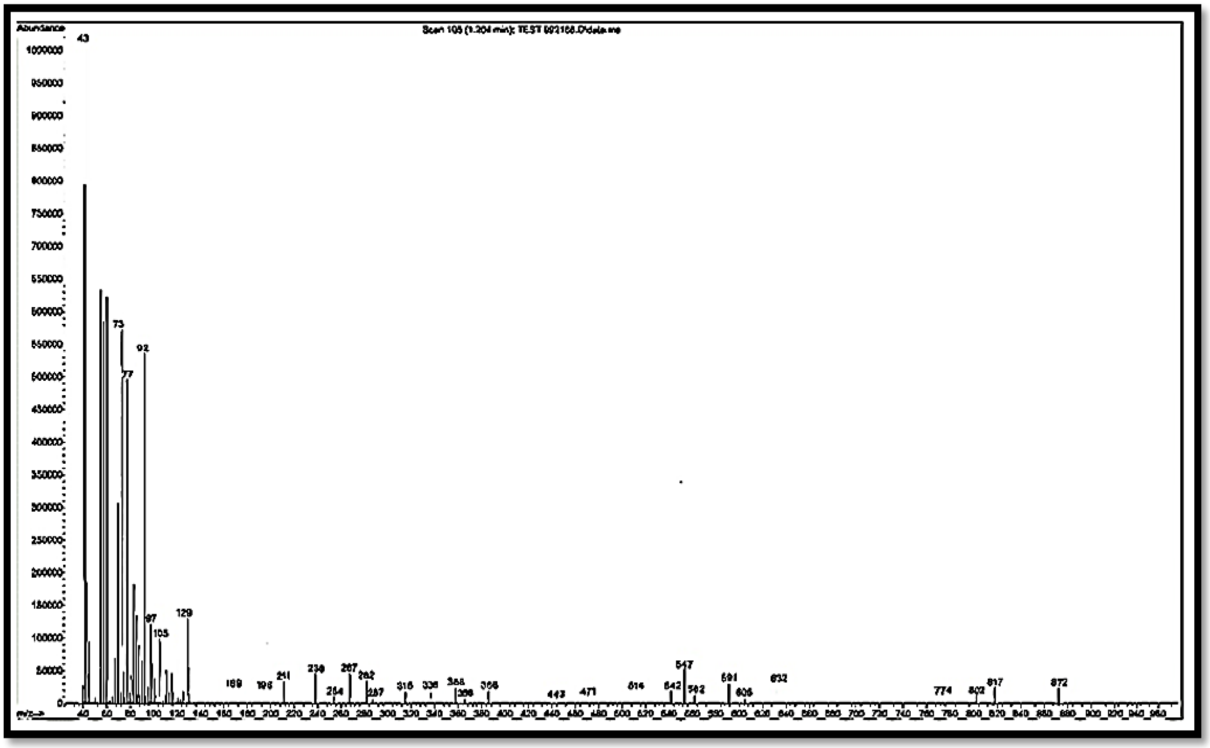

Figure 22: Mass spectrometry of amide derivative A6.

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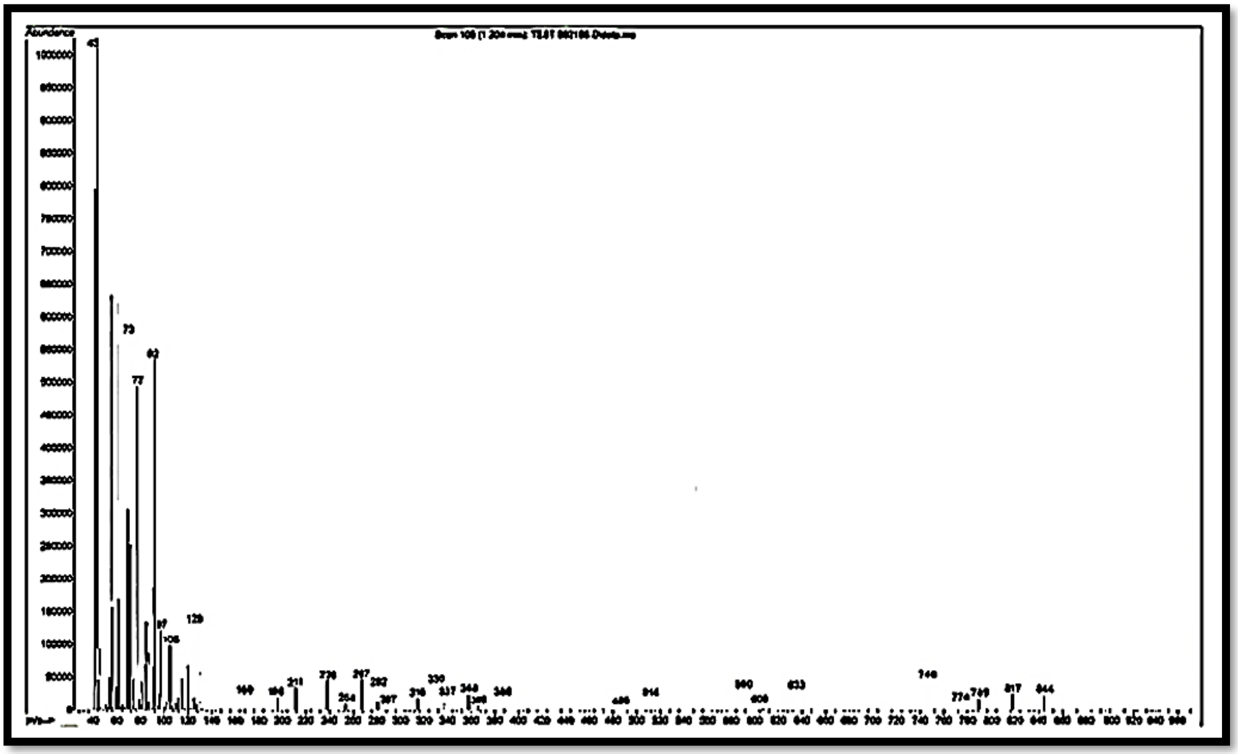

Figure 23: Mass spectrometry of amide derivative A7.

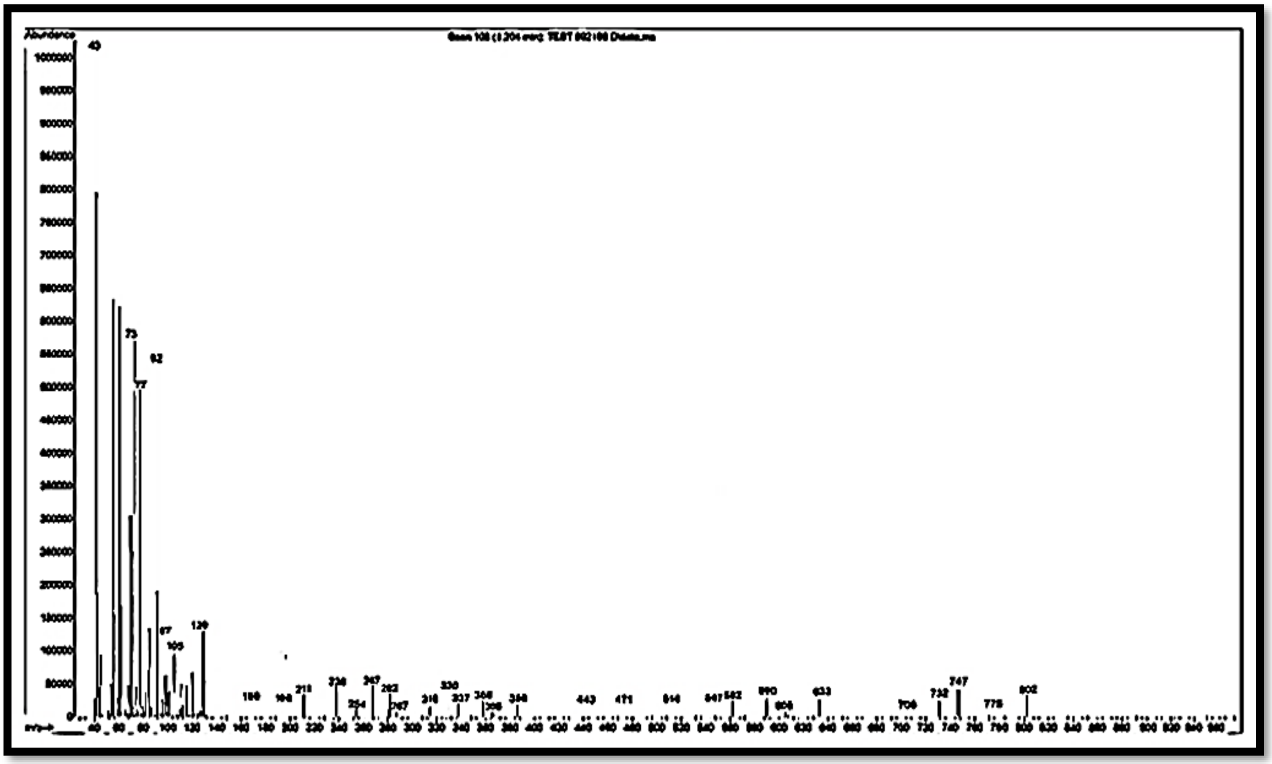

Figure 24: Mass spectrometry of amide derivative A8.

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4. Conclusions

New amide derivatives have been synthesized by direct reaction between 3, 3'-(1, 4-phenylene) bis (1-(4-aminophenyl) prop-2-en-1-one) containing an amine group with mono and dicarboxylic acids in the presence of sodium hydroxide under reflux. All synthesized compounds are given a good yield without using a catalyst.

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تحضير وتشخيص بعض مشتقات الأمايد الجديدة لمركب

3,3'-(1,4-Phenylene) Bis (1-(4-Aminophenyl) Prop-2-en-1-one)

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المستخلص

تم تحضير سلسلة جديدة من مشتقات الأميد [A1–A8] في حصيلة جيدة جداً عن طريق التصعيد التكثيفي المباشر لمركب bis-chalcone [3,3'-(1,4-phenylene) bis (1-(4-aminophenyl) prop-2-en-1-one)] مع أحماض كربوكسيلية مختلفة وأكريلاميد في وجود مذيب مناسب وكمية من هيدروكسيد الصوديوم (NaOH). شخصت تراكيب الأمايد المحضرة بتقنيات طيفية مختلفة، بما في ذلك (التحليل الطيفي بالأشعة تحت الحمراء FTIR)، و (التحليل الطيفي بالرنين المغناطيسي النووي البروتوني ^{1}H-NMR)، والتحليل الطيفي الكتلي، وأكدت الأطياف الناتجة جميعها البنية المتوقعة لمشتق ثنائي الجالكون المحضر.

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