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Electrical properties of amino acid substituted novel cinnamic acid compounds

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ABSTRACT

Amino acid conjugates are combinations of one or more compounds, at least one of them active in terms of biological, pharmacological or physical. In this work, some novel amino acid conjugates, four different aldehydes (Pyrene, Naphthalene, Phenanthrene and Phenyl benzyl) have been chosen and from these initial materials four different amino acid conjugates were synthesized. The compounds from initial steps to final steps have been characterized by ^{1}H and ^{13}C APT NMR spectroscopy. The dielectric measurements were done by QuadTech 7600 LRC impedance analyzer. The dielectric constant (ε′) dielectric loss (ε″) and ac conductivity (σ) values of amino acid conjugates in the frequency of 1 kHz and at 25 °C. The results show that the increase in conjugation affect the dielectric constant and conductivity.

1. Introduction

Amino acid/peptide conjugates are named because of covalently bonded of amino acid from one of the active functional side to different group of compound [1–5]. In this work, the group that is attached to the amino acid amino terminus is cinnamic acid derivatives [6–9]. The aim of this work is to synthesize novel amino acid conjugates via benzotriazole methodology and evaluate dielectric properties such as dielectric constant, dielectric loss and conductivity based on structure activity relationship. In here, four different cinnamic acid compounds were synthesized according to Doebner modification [10] and carboxylic acid side was activated by 1*H*-Benzotriazole in order to react with free amino acid glycine. Four different amino acid conjugates were obtained and dielectric properties were measured. The obtained results were discussed in discussion section. (see Table 1, Scheme 1)

Cinnamic acid derivatives (CADs) are a class of natural substances that found in flowers, vegetables, fruits and used as phenolic compounds which plays important role for the production of various pharmaceutical elements [11,12]. Numerous works have been studied about cinnamic acid compounds based on synthesis and biological activity, however; the lack of adequate work in terms of physical properties for amino acid conjugates will make the result of this work more important for scientifically and technologically. The cinnamic acid compounds exhibit numerous pharmacological effects such as antimalarial [13], antifungal [14], antituberculotic [15], antimicrobial [16], anticancer and antioxidant [17]. Four different ring size (two, three and four fused benzene rings) were used except cinnamic acid itself for this work in order to compare and evaluate structure-activity relationship in terms of electrical properties.

Benzotriazole methodology has been developed after A.R. Katritzky's enormous works since 1985 [18]. In this work, benzotriazole methodology successfully used to obtained amino acid conjugates which are differ from aromatic conjugation numbers. The reason why benzotriazole preferred is that it is inexpensive, stable, nontoxic and require mild reaction conditions both removing and introducing steps of the reaction [19]. It is a synthetic auxiliary which activates carboxylic acids to be enable to react with various nucleophilic groups. Benzotriazole derivatives are used as corrosion inhibitors, supramolecular ligands, dyes, potential drug candidates, fluorescent and radiochemical markers. Benzotriazole is preferred in organic synthesis due to its odorless, non-toxic, stable properties, long shelf life and solubility in various organic solvents. It is used in many organic reactions due to its following characteristics: better leaving group than halogens, having both electron donor and electron releasing feature and stabilizing carbonations [20].

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Table 1
The dielectric constant (ε') dielectric loss (ε'') and ac conductivity (σ) values of amino acid conjugates in the frequency of 1 kHz and at 25 °C.

Sample	ϵ'	ϵ''	$\log\sigma_{ac}$ (S/cm)
Phenant-CA-Gly-OH	7,84	0.170	−9,56
Pyrene-CA-Gly-OH	6,72	0.088	−9,50
Phen-Benzyl-CA-Gly-OH	4,67	0.082	−9,74
Naft-CA-Gly-OH	3,20	0.027	−9,57

Limited number of studies have been found in the literature about physical properties of amino acid conjugates [21,22]. On the other hand, no works were found for dielectric measurements of amino acid conjugates.

In this work, benzotriazole mediated cinnamic acid derivatives were synthesized and reacted with non-protected glycine amino acid to get four amino acid conjugates. All compounds from initial steps to final steps have been characterized by ^{1}H and ^{13}C APT NMR spectroscopy. The dielectric measurements were done by QuadTech 7600 LRC impedance analyzer.

2. Experimental

2.1. Materials and methods

4-Phenylbenzaldehyde, 1-naphthaldehyde, 9-Phenanthrenecarboxaldehyde and 1-Pyrenecarboxaldehyde were purchased from Sigma Aldrich, Alfa Aesar, and Across, respectively. Piperidine, Thionyl chloride and Pyridine were supplied from Sigma-Aldrich. Triethylamine, benzotriazole reagents and ethanol (EtOH), acetonitrile (MeCN), n-hexane, and dichloromethane (DCM) solvents were obtained from Merck. ^{1}H, and ^{13}C NMR spectra of the compounds were analyzed using a Bruker DPX-400 spectrometer. For the NMR studies, DMSO-d$_6$was used as the solvent for these syntheses. The dielectric parameters were carried out on the pellets prepared by applying five tons of pressure. The dielectric analysis was recorded using a QuadTech 7600 LRC impedance analyzer from to 100 Hz to 20 kHz frequencies.

2.2. Synthesis

2.2.1. General procedure for cinnamic acid derivatives

Aldehyde derivatives (1 eq.), malonic acid (1.1 eq.), pyridine (5.7 eq.) and piperidine (0.2 eq.) were added to a round bottom reaction flask and reflux overnight. After reaction was cooled to room temperature, the mixture was pour into beaker which contained sufficient amount of water and stirred until pH of the new mixture stayed around 5 b y using 4 N HCl. Obtained precipitated was filtered and wash with water. Desired product was obtained as solid (Yields 75–95%) [10].

2.2.2. Synthesis of (E)-3-(pyren-4-yl)acrylic acid **(pyrene-CA-OH)**

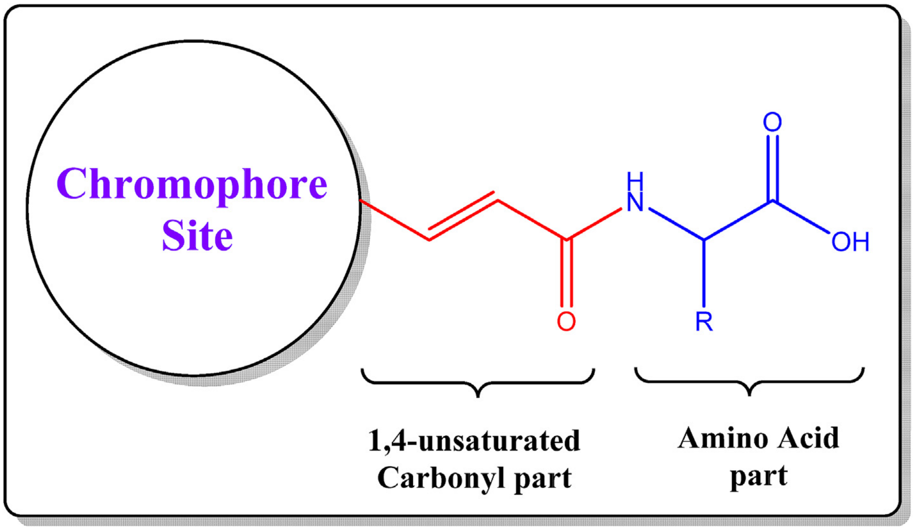
Yellow solid. Yield %75 mp:^{1}HNMR (DMSO-d_6) δ: 12.61 (1H, s), 8.74–8.70 (1H, d, J = 15.6 Hz), 8.57–8.53 (2H, t, J = 8.8 Hz), 8.40–8.37 (2H, dd, J = 3.2 Hz), 8.35–8,33 (2H, d, J = 8.8 Hz), 8.29–8.22 (2H, dd, J = 9.2 Hz), 8.15–8.12 (1H, t, J = 7.6 Hz), 6.87–6.83 (1H, d, J = 15.6 Hz).^{13}C-APT NMR (DMSO-d_6): 168.10, 140.36, 132.58, 131.32, 130.67, 129.39, 129.20, 128.96, 128.33, 127.84, 127.12, 126.61, 126.12, 126.35, 125.81, 125.12, 124.51, 122.78, 122.13.

2.2.3. Synthesis of (E)-3-(naphthalen-1-yl)acrylic acid **(Napth-CA-OH)**

White solid. Yield %95 mp:^{1}H NMR (DMSO-d_6) δ: 12.58 (1H, s), 8.42–8.38 (1H, d, J = 15.6 Hz), 8.23–8.21 (1H, d, J = 8.4 Hz), 8.04–7.95 (3H, m), 7.67–7.56 (3H, m), 6.64–6.60 (1H, d, J = 15.6 Hz)^{13}C-APT NMR (DMSO-d_6): 168.33, 143.44, 142.08, 139.75, 133.99, 129.49, 129.25, 128.37, 127.54, 127.15, 120.10.

2.2.4. Synthesis of (E)-3-(phenanthren-9-yl)acrylic acid **(Phenant-CA-OH)**

Green solid. Yield %80 mp:^{1}H NMR (DMSO-d_6) δ: 8.94–8.91 (1H, dd, J = 2,4 Hz), 8.86–8.84 (1H, d, J = 8 Hz), 8.42–8.38 (1H, d, J = 15.6 Hz), 8.31 (1H, s), 8.24–8.23 (1H, d, J = 2 Hz), 8.09–8.08 (1H, d, J = 8 Hz), 7.79–7.69 (4H, m), 6.73–6.69 (1H, d, J = 15.6 Hz). ^{13}C-APT NMR (DMSO-d_6): 168.09, 140.87, 131.33, 130.87, 130.67, 129.95, 129.73, 128.35, 127.90, 127.70, 127.62, 126.85, 124.46, 124.06, 123.33.



Scheme 1. General representation of Amino acid conjugate.

2.2.5. Synthesis of(E)-3-([1,1′-biphenyl]-4-yl)acrylic acid(**Phenybenzyl-CA-OH**)

Light grey solid. Yield %85 mp:^{1}H NMR (DMSO-d_6) δ: 7.79–7.77 (2H, d, J = 8.4 Hz), 7.74–7.72 (4H, dd, J = 2.4, 1.2 Hz), 7.66–7.62 (1H, d, J = 16 Hz), 7.51–7.47 (2H, t, J = 15.2 Hz), 7.42–7.38 (1H, t, J = 14.8 Hz), 6.61–6.57 (1H, d, J = 16 Hz). ^{13}C-APT NMR (DMSO-d_6): 168.33, 143.44, 142.08, 139.75, 133.99, 129.49, 129.25, 128.37, 127.54, 127.15, 120.27.

2.2.6. General procedure for amino acid conjugates

Amino acid (1.1 eq.) was dissolved in acetonitrile and water (7:3) and tertiary amine base such as triethylamine or diisopropylethylamine (1.2 eq.) added to the reaction flask. After the dissolving of all amino acid, N-acylbenzotriazol (1.0 eq.) was added to the mixture and stirred at room temperature for overnight. The reaction was monitored by thin layer chromatography. After completion of reaction, the solvent of the mixture was removed and the residue precipitated in water. Desired product was obtained after filtration as solid (yield 60–75%).

2.2.7. Synthesis of(E)-(3-(pyren-4-yl)acryloyl)glycine **(pyrene-CA-Gly-OH)**

Dark green solid. Yield %60 mp: ^{1}H NMR (DMSO-d_6) δ: 3.95–3.93 (2H, d, J = 5.2 Hz, H^{13}), 7.12–7.08 (1H, d, J = 15.6 Hz, H^{11}), 8.11–8.08 (1H, t, J_1 = 7.6 Hz and J_2 = 7.6 Hz, H^{10}), 8.17–8.15 (1H, d, J = 8.8 Hz, H^9), 8.23–8.20 (1H, d, J = 8.8 Hz, H^1), 8.38–8.26 (5H, m, $H^{2,3,6,7,8}$) and 8.57–8.53 (3H, m, $H^{4,5,12}$). ^{13}C-APT NMR (DMSO-d_6): 169.03, 165.03, 145.93, 135.65, 132.03, 131.37, 130.74, 129.34, 129.13, 128.90, 128.58, 127.82, 127.05, 126.39, 126.12, 125.83, 125.37, 124.61, 124.34, 122.97, 42.16.

2.2.8. Synthesis of (E)-(3-(naphthalen-1-yl)acryloyl)glycine **(Napth-CA-Gly-OH)**

Light yellow solid. Yield %75 mp:^{1}H NMR (DMSO-d_6) δ:3.95–3.96 (2H, d, J = 5,6 Hz, H^{11}), 6.78–6.82 (1H, d, J = 15.6 Hz, H^9), 7.57–7.65 (3H, m, $H^{2,6,8}$), 7.83–7.81 (1H, d, J = 7.2 Hz, H^5), 8.02–8.00 (2H, d, J = 8.4 Hz, $H^{4,7}$), 8.27–8.20 (2H, m, $H^{1,3}$) and 12.69 (1H, s, H^{12}). ^{13}C-APT NMR (DMSO-d_6): 171.77, 165.71, 136.17, 133.82, 132.38, 131.29, 130.13, 129.15, 127.40, 126.72, 126.24, 125.15, 125.00, 123.64, 41.40.

2.2.9. Synthesis of (E)-(3-(phenanthren-9-yl)acryloyl)glycine **(Phenant-CA-Gly-OH)**

Light green solid. Yield %75 mp:^{1}H NMR (DMSO-d_6) δ: 3.89–3.87 (2H, d, J = 5.2 Hz, H^{13}), 6.98–6.94 (1H, d, J = 15,6 Hz, H^{11}), 7.76–7.64 (5H, m, $H^{2,3,6,7,9}$), 8.05–8.03 (1H, d, J = 7.6 Hz, H^{10}), 8.12 (1H, s), 8.25–8.22 (2H, t, J_1 = 8 Hz J2 = 7.6 Hz), 8.31(1H, s), 8.83–8.81 (1H, d, J = 8.4 Hz, H^5), 8.90–8.88 (1H, d, J = 7.2 Hz, H^4). ^{13}C-APT NMR (DMSO-d_6): 185.58, 173.11, 165.25, 145.97, 136.22, 131.67, 131.40, 130.17, 129.47, 128.02, 127.78, 127.67, 127.56, 126.47, 125.98, 124.63, 124.01, 123.31, 43.14.

2.2.10. Synthesis of (E)-(3-([1,1′-biphenyl]-4-yl)acryloyl)glycine **(Phenybenzyl-CA-Gly-OH)**

white solid. Yield %70 mp:^{1}H NMR (DMSO-d_6) δ:3.81–3.80 (2H, d, J = 5.2 Hz, H^9), 6.98–6.94 (1H, d, J = 16 Hz, H^7), 7.38–7.36 (1H, d, J = 7.2 Hz, H^{16}), 7.49–7.42 (3H, m, $H^{1,3,3'}$), 7.67–7.65 (6H, d, J = 9,6 Hz, $H^{2,2',4,4',5,5'}$) and 8.14–8.12 (1H, t, J_1 = 4.8 Hz and J_2 = 5.2 Hz, H^8). ^{13}C-APT NMR (DMSO-d_6):168.53, 165.24, 141.35, 139.84, 138.47, 134.64, 129.47, 128.66, 127.56, 127.07, 122.96, 43.31.

3. Result and discussion

3.1. Synthesis and characterization

All *trans*-cinnamic acids were synthesized using the Knoevenagel–Doebner condensation between aromatic benzaldehydes and malonic acid in the presence of piperidine as a catalyst [10].

In each target compound, the presence of $-CH_2$ protons from the glycine and olefinic protons from the 1.4 unsaturated conjugate portion of cinnamic acid indicates that the compounds have been successfully obtained. There are characteristic peaks that indicate that the targeted structures are formed when the NMR spectra of the compounds are examined. The chromophore portions of the target compounds contain aromatic protons and carbon atoms. However, the presence of aliphatic and olefinic protons and carbons in the NMR spectra proves that clearly targeted amino acid conjugates are formed. When the ^{1}H and ^{13}C APT NMR spectrum of the Naft-CA-Gly-OH is examined, the carboxylic acid −OH proton is resonant at 12.69 ppm, the glycine $-CH_2$ protons appear as doublet at 3.96–3.95, and the olefinic CH protons showed up as doublet at 6.78–6.82 ppm and the J constant of the olefinic = CH is 15.6 Hz indicates that the structure is trans.

The carboxylic acid carbonyl peak at 171.71 ppm, the amide carbonyl carbon at 165.71 ppm and the aliphatic carbon amino acid portion at 41.40 ppm are successfully interacting with cinnamic acid. Phenant-CA-Gly-OH and Pyrene-CA-Gly-OH compounds are similar in structure, but when NMR spectra are evaluated, characteristic protons and carbon atoms are slightly resonant in different areas. Olefinic and aliphatic protons and carbons, along with carbonyl carbons, help us to understand that structures are formed so that $-CH_2$ aliphatic protons are split as doublet in both compounds resonating at 3.89–3.87 and 3.94–3.93 ppm, respectively (see Scheme 3 and 4).

Olefinic protons appear to be around 6.78–6.82 and 7.08–7.12 ppm. The J constant of the olefinic protons in both structures is 15.6 Hz, which is proof that the structuresare in a trans position. The aliphatic carbon atoms of the Phenant-CA-Gly-OH and Pyrene-CA-Gly-OH were resonated at 43.14 and 42.15 ppm. The ^{1}H NMR spectra of Naft-CA-Gly-OH and Pyrene-CA-Gly-OH in Schemes 3 and 4.

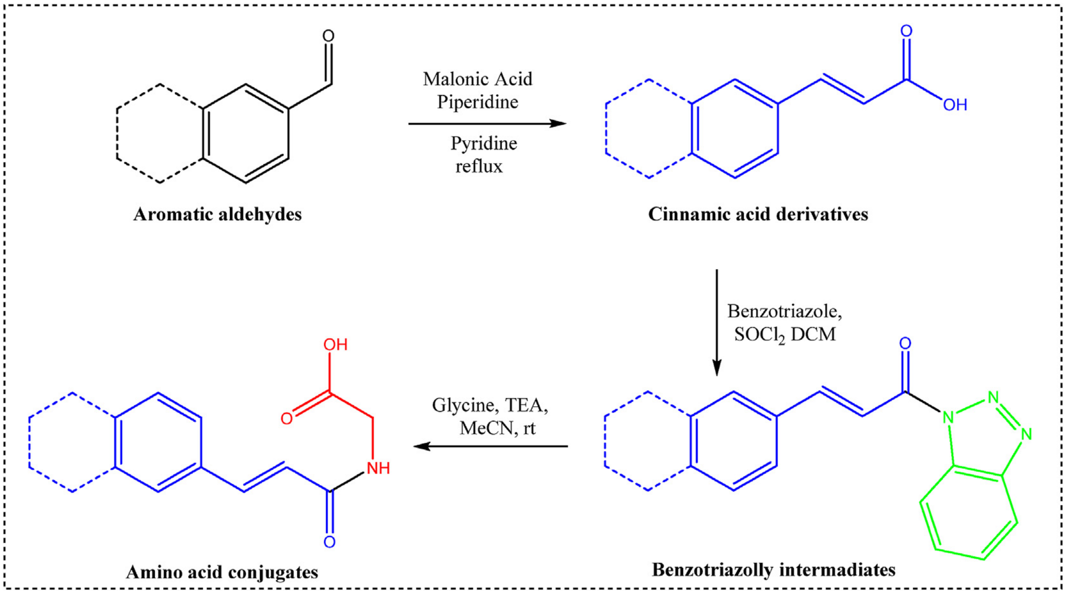
3.2. Dielectric measurements

When a material is capable of storing energy when an external electric field is applied, it is referred to as "dielectric."The dielectric constant indicates how much energy is stored in the external electric zone under the influence of an area and how much energy is lost in the material.

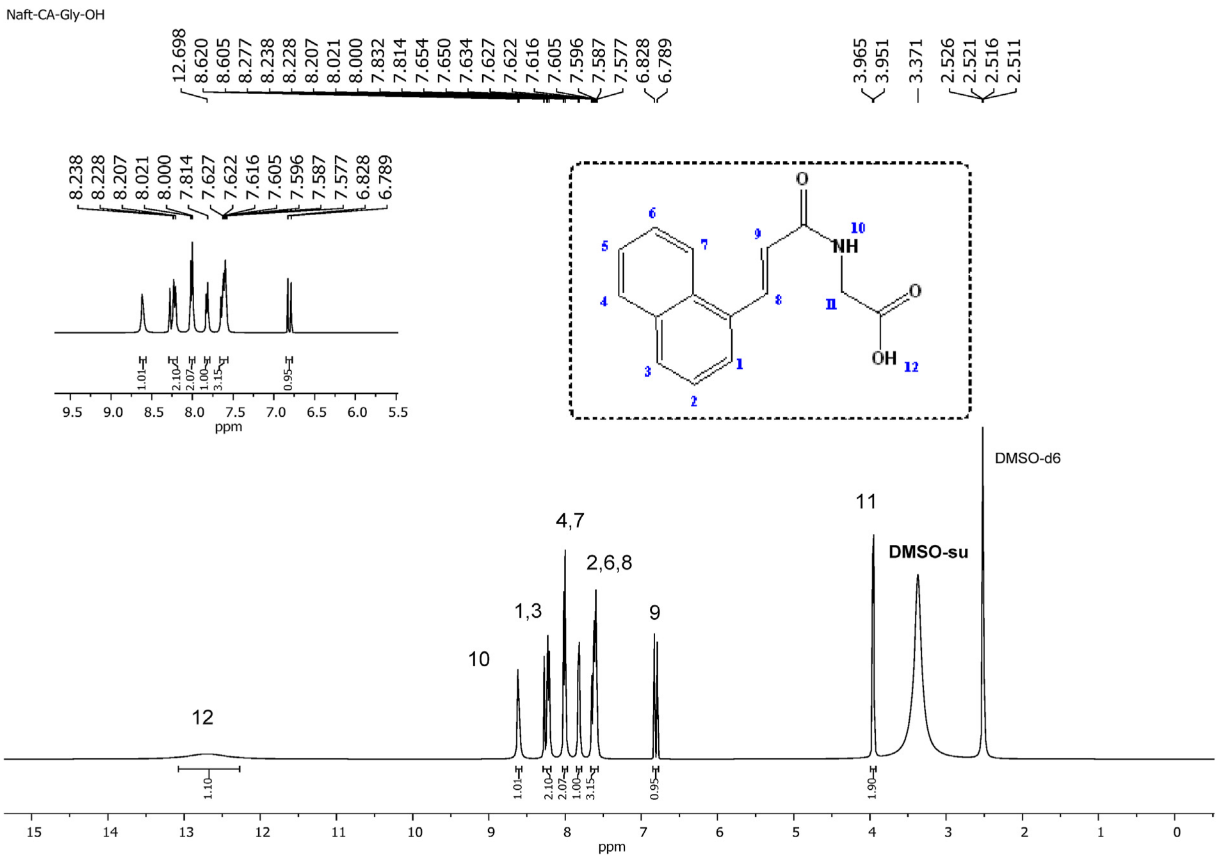
Under the influence of the electric field, electrons and atoms are displaced. As a result, the electrical charge centers shift and electrical polarization occurs. The resulting electrical dipoles provide the accumulation of electrical charge on the surface of the dielectric material. Polarization is the reaction of the material to the electric field [23,24].

Dielectric losses depend on the types of polarization. Dielectric losses due to surface charge polarization occur around 104 Hz, those resulting from dipole polarization occur around 108 Hz, losses due to atomic polarization occur around 1012 Hz, and dielectric losses due to electronic polarization occur around 1016 Hz. While dielectric losses increase with rising temperature in materials with atomic and electronic polarization, the dielectric constant does not change much at low temperatures.

The dielectric constant is the expression of a polarization-specific measurement. Variation of dielectric properties; is a

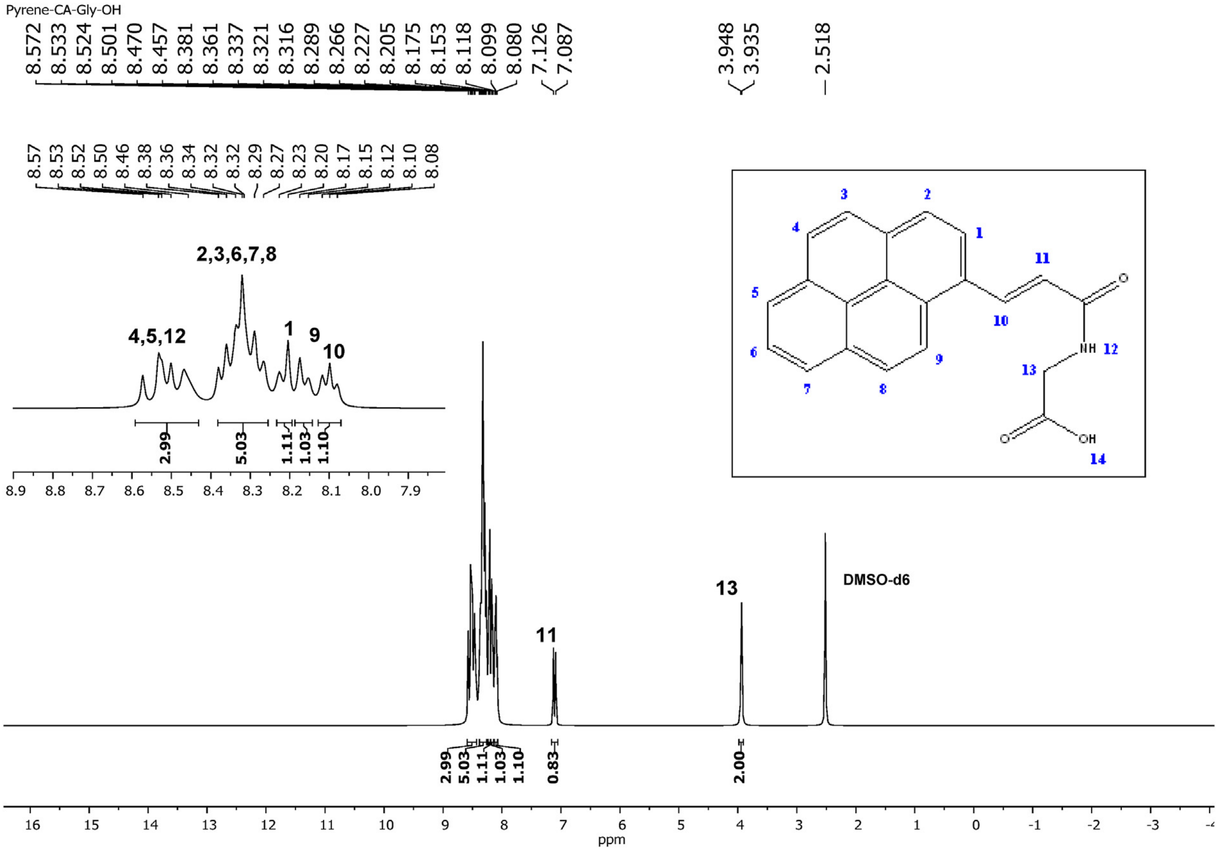


Scheme 2. General synthetic route for amino acid conjugates.

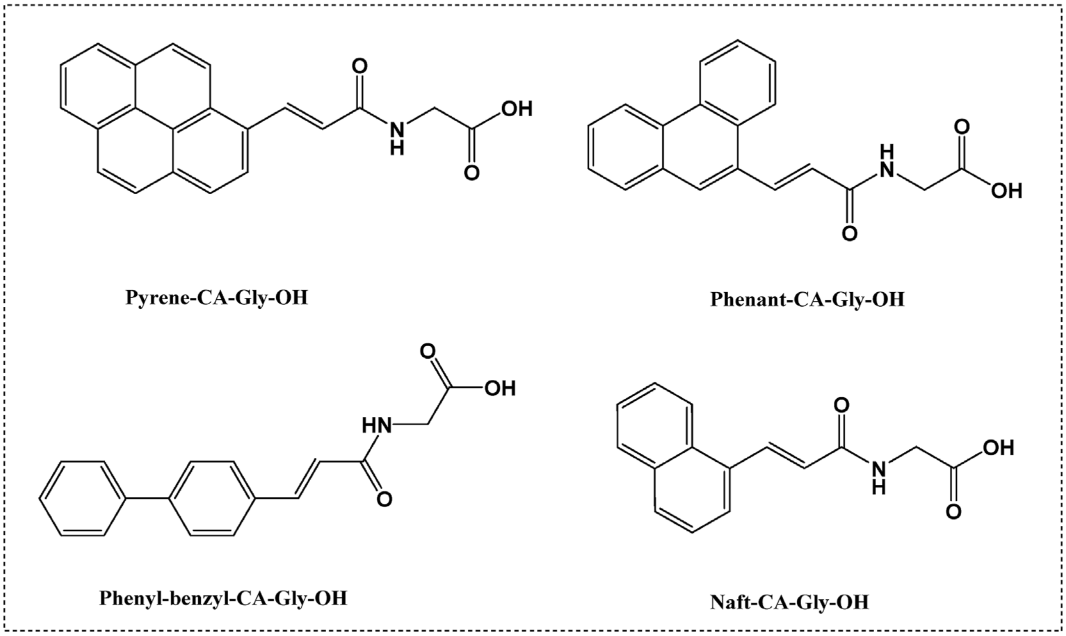


Scheme 3. The ^{1}H NMR spectrum of Naft-CA-Gly-OH.

result of electronic, ionic and molecular polarizability. These properties are related to the physical and chemical structures of the compounds. In order to investigate the dielectric behavior of the synthesized compounds, the sample area (A) calculated by composing the compounds under 4 tons' pressure. For the area calculation, sample thicknesses are measured in a device measuring thickness of 0.001 mm, and the area of each plate is: A (area of the sample) = π.t. $(d/2)^2$. Then, parameters such as capacitance values (Cp), dielectric loss factor (DF) and conductivity (Gp) measured by gold electrodes with Impedance analyzer and these parameters were calculated by the following equations (1) and (2) [25].



Scheme 4. The ^{1}H NMR spectrum of Pyrene-CA-Gly-OH.



Scheme 5. Structure of analyzed compounds.

$$\varepsilon' = C_p \frac{d}{A\varepsilon_0} \tag{1}$$

$$\varepsilon'' = \varepsilon' \mathbf{DF} \tag{2}$$

where ϵ' is dielectric constant, ϵ_0 is the dielectric constant of vacuum (8.854×10^{-12}), d is the thickness (m) and A is the effective

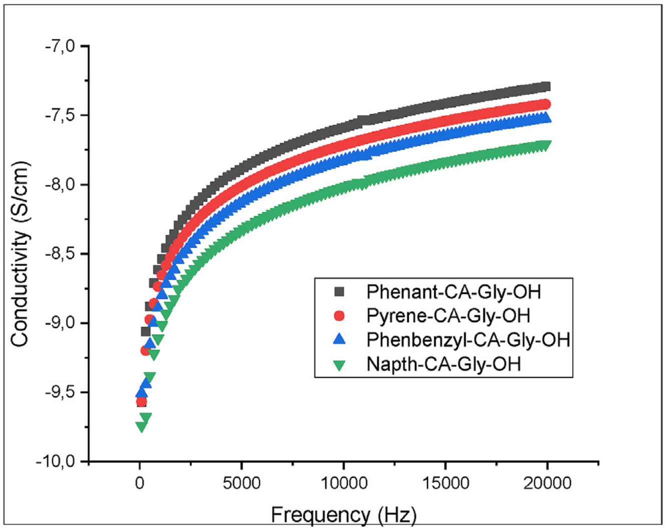


Fig. 1. Dependence of ac conductivity of samples as a function of frequency.

area (m^2) of the sample and C is the capacitance (F) of the test device.

And then the AC conductivity was calculated below equation (3).

$$\sigma = G_p \frac{d}{A} \tag{3}$$

where σ is ac conductivity, d is the thickness (m) and A is the effective area (m^2) of the sample.

AC (σ_{ac}, Scm^{-1}) conductivity is crucial for the transmission mechanism. The conductance measurements of the compounds were analyzed at between 1 Hz and 20 kHz based on the alternating current (AC) conductivities. Fig. 1 shows the conductivity behavior of selected compounds versus frequency at room temperature. The conductivity values change with the frequency due to the electrical conductivity of the compounds depends on mobility of electrons or ions in their structures [26–28].

In the AC measurement process, the sample is subjected to an amplitude AC voltage, which can be considered relatively small, the AC current corresponding to this current signal is recorded, and the change between the current and voltage is recorded as a function of time. The amplitude of the signal used in this process is very important. It must be small enough to cause the system to react linearly, but large enough to be recorded [29–31]. In Fig. 2 shows that ε′ and ε″ values decreased with increase in frequency because of polarization effects. At low frequencies, orientation and interface polarizations are more influential. Therefore, the dielectric loss factor is high. Another important point is that as conjugation in molecules increases, the dielectric constant and dielectric loss values were getting higher but anthracene bearing conjugate which carries 3 benzene ring shows higher dielectric constant than four member pyrene bearing conjugate that might be caused by the longer way that electron travels in Table 1. The aromatic ring caused the structure to be polar. So, all target compounds have same amino acid (glycine) part which contribute equally to the polarization but the number of aromatic ring varies and that make significant difference on the dielectric values. Compare to reported studies, amino acid conjugates shows higher conductivity. In similar studies in the literature, when the conductivity values obtained for chalcone compounds are compared with amino acid conjugates, the highest conductivity value in chalcone compounds was 4.97 10 -9 S/cm, while this value was 4.45 × 10 -9 S/cm in conjugates. This shows that the increase in the number of conjugation increases in direct proportion to the conductivity [32,33].

4. Conclusion

Collectively, the amino acid conjugates have a very important place in chemistry and our work will contribute to physical chemistry section which has limited number of related works. Free glycine was reacted with four aromatic *N*-benzotriazolyl cinnamic acids and all conjugates were successfully characterized by ^{1}H and ^{13}C APT NMR spectroscopy and evaluated in terms of dielectric constant, dielectric loss and conductivity. The dielectric constant behavior of compounds was altered depend on frequency in which it is getting higher with low frequencies. The reason of such behavior is due to space charge, orientation, electronic and ionic polarization and lower dielectric values were caused most likely to the loss of these polarizations. The three benzene ring containing conjugates (Phenant-CA-Gly-OH) shows highest dielectric constant (ε′) 7.84, on the other hand, naphthalene bearing conjugates (Naft-CA-Gly-OH) have the lowest value of 3.20 which shows that the increase in the ring number has direct effect to dielectric properties. These results indicate that three and four-member ring containing conjugates could be used for optoelectronic and other related applications.

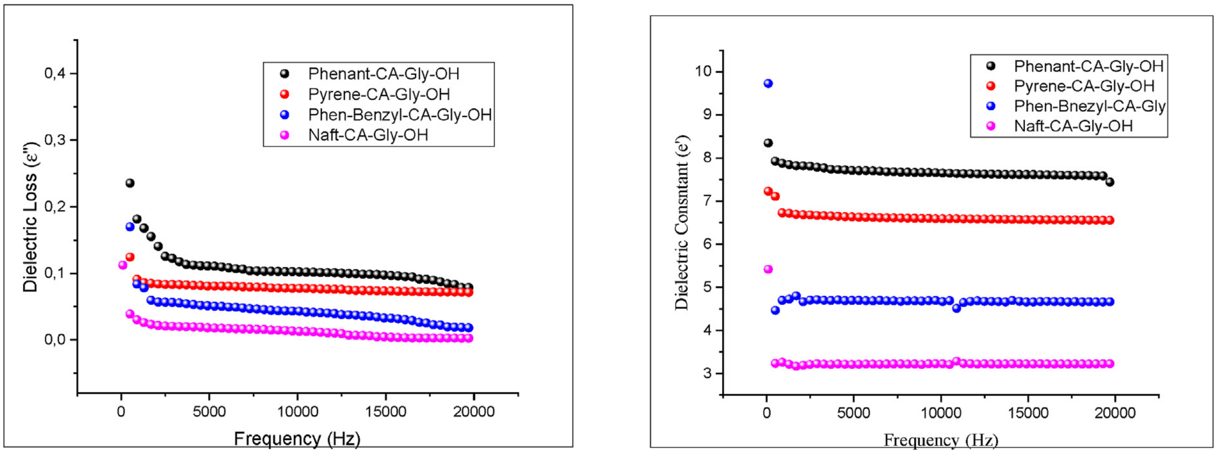


Fig. 2. Dependence of dielectric loss anddielectric constant of samples as a function of frequency.

Author credit statement

EC, AOG and AÇ conceived and designed the research. KK, carried out NMR and dielectric experiment. EC carried out all experiments. EC, KK, AOG and AÇ analyzed the data and wrote the manuscript

Declaration of competing interest

Author declare that there is no conflict of interest.

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