JOURNAL OF SCIENCE
SAKARYA UNIVERSITY

Sakarya University Journal of Science

ISSN 1301-4048 | e-ISSN 2147-835X | Period Bimonthly | Founded: 1997 | Publisher Sakarya University
http://www.saujs.sakarya.edu.tr/en/

Title: Synthesis and Characterisation of Polyaromatic Chalcones with Electron Donation

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Recieved: 2019-07-20 16:43:02

Accepted: 2020-01-09 14:09:40

Article Type: Research Article
Volume: 24
Issue: 2
Month: April
Year: 2020
Pages: 347-356

How to cite
Alparslan Atahan; (2020), Synthesis and Characterisation of Polyaromatic Chalcones with Electron Donation. Sakarya University Journal of Science, 24(2), 347-356, DOI: https://doi.org/10.16984/saufenbilder.594611
Access link
http://www.saujs.sakarya.edu.tr/en/issue/52471/594611

Sakarya University Journal of Science 24(2), 347-356, 2020

Synthesis and Characterisation of Polyaromatic Chalcones with Electron Donation

Alparslan ATAHAN*[1]

ABSTRACT

In this study, a series of chalcone derivatives was successfully synthesized via condensation of 1-acetylpyrene with dimethylamine, diphenylamine or carbazole containing benzaldehyde derivatives at basic conditions. Spectral characterisations were acquired by 1 and 2 dimensional NMR techniques and FTIR. In addition, UV-vis and thermal analysis studies were performed to determine their absorption properties and thermal behaviours, respectively. Lastly, surface and film generation properties were investigated by the means of SEM images on ITO glass to determine usability potential in organic electronics.

Keywords: Chalcone, Pyrene, Triphenylamine, Carbazole, Characterisation.

1. INTRODUCTION

Chalcones which have 1,3-diaryl-2-propen-1-one skeleton are a family of aromatic ketones and have huge fascination due to their simple chemistry and pervasive applications in medicinal [1,2], and material chemistries [3,4]. In addition, they are quite functional as chemical intermediates in the synthesis of many significant compounds such as quinolines [5], pyrimidines [6], aza-BODIPY's [7], chromones [8], and oxazoles [9] etc.

As a bioactive molecule, it is well known that natural and synthetic chalcone derivatives exhibit wide spectrum of chemotherapeutic properties such as antimicrobial [10], antioxidant [11], anticancer [12], enzyme inhibitor [13], anti-inflammatory [14], antimalarial [15] and more. These mentioned properties can be attributed to their linear and nearly planar structure and extended conjugation.

Alongside of their broad-spectrum bioactivity and importance in synthetic chemistry, they have characteristic electronic structure due to conjugation and donor-acceptor structure and this property makes them functional compounds in photophysics [16] as well. In this context, they have a wide range of applications as promising materials for non-linear optics [17], also in electro-active fluorescence [18]. In addition, chalcones are useful compounds as fluorescent sensors for the recognition of some cations [19].

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Chalcone structures are easily prepared via Claisen-Schmidt condensation between acetophenone and benzaldehyde or their substituted derivatives [20]. In the same way, their polyaromatic (i.e., anthracene, phenathrene, pyrene) counterparts can be obtained via replacing one (or two) ring(s) by a polyaromatic group, generating the so-called polyaromatic chalcones. These structures are commonly prepared by systematic chemical modifications via condensation using polyaromatic aldehyde and required substituted acetophenone or vice versa [21, 22]. The addition of a polyaromatic scaffold into chalcone compounds can bring about significant improvement in their physical properties such as absorption, fluorescence, and beneficial sensing capacity against cations, anions, or some molecules [23-25]. It is believed that these improved performances depend on the chemical unification of these electronically specific groups in the chalcone structure.

In this context, pyrene is a well-known polyaromatic hydrocarbon and has also quite interesting properties including high fluorescence [26], high optical contrast [27], and white-light emission [28]. Moreover, various pyrene derivatives exhibit surprising exciplex emission at higher concentrations or solid state [29,30]. In other words, π-stacking or intermolecular interaction of among pyrene rings can change whole photophysical characteristics and this phenomenon promotes an increase or quench at emission.

Since the promising potential of mentioned structures in significant areas, it is important to report novel compounds and their critical properties. Taking into account this fact and our previous studies [31,32], herein, the objective has been to generate polyaromatic chalcones which are containing various electron donating groups. In order to accomplish this objective, mentioned polyaromatic chalcones have been successfully prepared and characterised by ^{1}H NMR, ^{13}C NMR, COSY, and FTIR. UV-vis studies and thermal gravimetric analyses have been performed to determine their absorption properties and thermal behaviours, respectively. Lastly, SEM images on ITO glass have been taken to determine surface and film generation properties which are important for usability in organic electronics.

In structural characterisation, proposed structures have been confirmed and *trans* conformations have been observed for all the target compounds. From UV-vis studies, it has been shown that the target compounds have a main absorption band between ~380-430 nm. In thermal characterisation, the target compounds were quite stable and masses were preserved up to about 350 ºC. Finally, SEM images showed that the compounds have good film generation properties on ITO glass.

2. MATERIALS AND METHODS

2.1 *General*

Chalcone compounds (**3a-c**) were synthesized via Claisen-Schmidt condensation at basic conditions. All the reagents and chemicals were purchased from Sigma-Aldrich**,** Merck or Fluka and they were AR grade with high purity. Used solvents were dried, distilled and purified according to the standard methods. Silica Gel TLC plates were used to monitor the reactions and crude products were purified by crystallization or silica gel column chromatography. NMR spectra were carried out by 400 MHz Bruker NMR Spectrometer in DMSO-d6. Chemical shifts were reported in ppm from tetramethylsilane ($(CH_3)_4Si$) for ^{1}H and ^{13}C. FTIR spectra were recorded by Shimadzu Prestige-21 spectrometer combined with an ATR system. Melting points were determined by Electrothermal IA-9200 apparatus. TGA analyses were performed Shimadzu DTG 60H - DSC 60 Thermal Analysis System. Absorption studies were realized by a PG Instruments T80 double beam spectrophotometer by using a quartz cells. FEI Quanta FEG 250 Scanning Electron microscope was used to get SEM images on ITO glass.

2.2 *Chemistry*

The target compounds were obtained as shown in Figure 1 followed by preparing starting compounds (1-acetylpyrene, 4-diphenylamino benzaldehyde, 4-(carbazol-9-yl)benzaldehyde) according to literature procedures [33-35].

Chemical structures were illuminated by ^{1}H-NMR, ^{13}C-NMR, COSY and FT-IR.

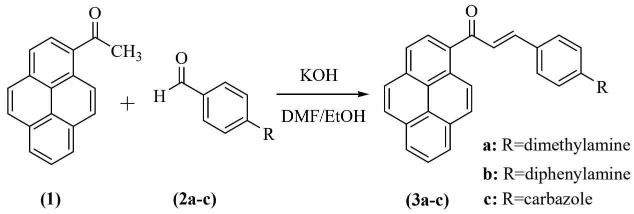

Figure 1. Synthesis of target compounds (3a-c)

2.2.1 *Synthesis of 1-acetyl pyrene*

Synthesis of 1-acetyl pyrene was accomplished according to the literature procedure [33]. Structure verification and purity control were realized by hydrogen NMR spectroscopy. M.p.: 172-173 ºC, yield: 65 %.

2.2.2 *Synthesis of 4-diphenylaminobenzaldehyde*

4-Diphenylaminobenzaldehyde was synthesized as reported in the literature procedure [34]. Structure verification and purity control were realized by hydrogen NMR spectroscopy. M.p.: 132-135 ºC, yield: 92 %.

2.2.3 *Synthesis of 4-(carbazol-9-yl)benzaldehyde*

4-(carbazol-9-yl)benzaldehyde was prepared as reported elsewhere [35]. Structure verification and purity control were realized by hydrogen NMR spectroscopy. M.p.: 154-157 ºC, yield: 87 %.

2.2.4 *Synthesis of chalcones (3a-c)*

The target chalcone compounds were successfully synthesized by Claisen-Schmidt condensation reaction as explained below. 0.122 g (0.5 mmol) of 1-acetylpyrene and 1.0 equi-molar amount of corresponding aldehyde were mixed in 20 mL of ethanol/DMF mixture (50/50 %, v/v) in the presence of 1.0 equivalent (0.028 g) KOH as catalyst and magnetically stirred at ambient temperature. After understanding the completion of the reactions by thin layer chromatography in three to five days, volatiles evaporated and resulting mixtures quenched with cold water. The precipitates were air-dried and re-crystallized in DCM/hexane or EtOAc/hexane to get pure chalcone products.

*(E)-3-(4-(dimethylamino)phenyl)-1-(pyren-1-yl)prop-2-en-1-one (**3a**)*

This target compound was reported previously [33]. ^{1}H NMR (400 MHz, DMSO-d$_6$) δ: 8.51 (d, *J*: 9,2 Hz, 1H), 8.41-8.26 (m, 7H), 8.14 (t, *J*: 7.6 Hz, 1H), 7.61 (d, *J*: 8,8 Hz, 2H), 7.51 (d, *J*: 15,6 Hz, 1H), 7.37 (d, *J*: 15,6 Hz, 1H), 6.71 (d, *J*: 8,8 Hz, 2H), 2.99 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ: 195.1, 152.6, 147.3, 135.2, 132.7, 131.3 (2C), 131.2, 130.6, 129.2, 129.1, 128.7, 127.8, 127.2, 126.6, 126.5, 126.2, 125.0, 124.9, 124.5, 124.2, 122.1, 122.0, 112.2(2C), 40.1(2C); FTIR: 3039, 2970, 1639, 1560, 1514, 1166, 840, 698 cm^{-1}; M.p.: 166-168 ºC; yield: 87 %.

*(E)-3-(4-(diphenylamino)phenyl)-1-(pyren-1-yl)prop-2-en-1-one (**3b**)*

^{1}H NMR (400 MHz, DMSO-d$_6$) δ: 8.54 (d, *J*: 9,2 Hz, 1H), 8.39-8.35 (m, 4H), 8.31-8.24 (m, 3H), 8.12 (t, *J*: 7.6 Hz, 1H), 7.64 (d, *J*: 8.4 Hz, 2H), 7.54 (d, *J*: 16.0 Hz, 1H), 7.49 (d, *J*: 16.0 Hz, 1H), 7.35 (t, *J*: 7.6 Hz, 4H), 7.14 (t, *J*: 7.6 Hz, 2H), 7.10 (d, *J*: 8.0 Hz, 4H), 6.86 (t, *J*: 8.4 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ: 195.2, 150.3, 146.6 (2C), 145.8, 134.5, 133.0, 131.2, 130.9 (2C), 130.6, 130.3 (4C), 129.5, 129.3, 128.9, 127.7, 127.6, 127.2, 126.9, 126.7, 126.4, 125.9 (4C), 125.1, 125.0 (2C), 124.9, 124.8, 124.5, 124.1, 120.9 (2C); FTIR: 3040, 2980, 1656, 1575, 1504, 1282, 842, 700 cm^{-1}; M.p.: 153-155 ºC; yield: 90 %.

(E)-3-(4-(9H-carbazol-9-yl)phenyl)-1-(pyren-1-yl)prop-2-en-1-one (3c)

^{1}H NMR (400 MHz, DMSO-d$_6$) δ: 8.69 (d, *J*: 9,2 Hz, 1H), 8.54 (d, *J*: 8.0 Hz, 1H), 8.47-8.30 (m, 6H), 8.26 (d, *J*: 8.0 Hz, 2H), 8.19-8.14 (m, 3H), 7.88 (d, *J*: 16.0 Hz, 1H), 7.81 (d, *J*: 16.0 Hz, 1H), 7.73 (d, *J*: 8.0 Hz, 2H), 7.49-7.44 (m, 4H), 7.32 (t, *J*: 6,4 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ: 195.0, 144.6, 140.2 (2C), 139.3, 134.0, 133.7, 133.4, 131.2, 131.1 (2C), 130.6, 129.8, 129.6, 129.2, 128.1, 127.8, 127.4, 127.3 (3C), 126.9, 126.8 (2C), 126.6, 125.0, 124.9, 124.5, 124.1, 123.5 (2C), 121.1 (2C), 120.9 (2C), 110.3 (2C);

FTIR: 3039, 2980, 1653, 1583, 1514, 1450, 1222, 831, 740 cm^{-1}; M.p.: 184-186 ºC; yield: 92 %.

2.3 *Absorption Studies*

All the absorption measurements were performed at 10^{-5} M concentration. For this, firstly, a certain amounts of **3a-c** were dissolved in 10 mL of each solvent to get 10^{-3} M solutions. 1:100 dilution of this stock solution gave 10^{-5} M solutions to be used for absorption spectra.

2.4 *Thermal Analysis*

Thermal properties were studied using 5-10 mgs of target compounds (3a-c). Thermograms were obtained in a platinum pan between room temperature and 850 ºC. The curves were obtained under nitrogen atmosphere with a flow rate of 100 mL/min. The heating rate was 10 °C/min.

2.5 *Surface Analysis Studies*

Target compounds were dissolved in dichlorobenzene at 1.0 M concentration and spin coated on appropriately cleaned ITO glass. After annealing on 150 °C plate for 60 seconds, SEM images were recorded to describe surface properties.

3. RESULTS AND DISCUSSION

3.1 *General*

The target compounds were successfully synthesized by using 1-acetyl pyrene and electron donation functionalized benzaldehyde derivatives. ^{1}H-NMR, ^{13}C-NMR, COSY, and FTIR analyses completely verified the proposed structures. All the target compounds were isolated with good yields (~90% levels).

3.2 *Structural Characterisation*

In ^{1}H NMR analysis, firstly, expected specific peaks of olefinic bond and electron donation group bonded phenyl ring on chalcone structures can be clearly seen. These signal systems can be easily selected by the means of signal shape, chemical shifts and coupling constants. For example, the AA’ signal systems of olefinic bond of **3a** can be seen as two doublets at δ = 7.35 and 7.55 with J = 15.6 Hz coupling constant. Like this, these signals for **3b** and **3c** can be seen at similar shape and chemical shifts (7.54, 7.49; J=16 Hz and 7.88, 7.81; J=16 Hz, respectively). In addition, phenyl ring signals associated with benzaldehyde can be seen as AA’BB’ system as written above.

On the other hand, pyrene related protons have been resonated usually as multiplets at lower magnetic fields due to lower electron densities. In ^{13}C NMR spectra, carbonyl carbon signals of all chalcones can be clearly seen at around δ = 190 ppm and other carbon signals are completely in accordance with proposed structures.

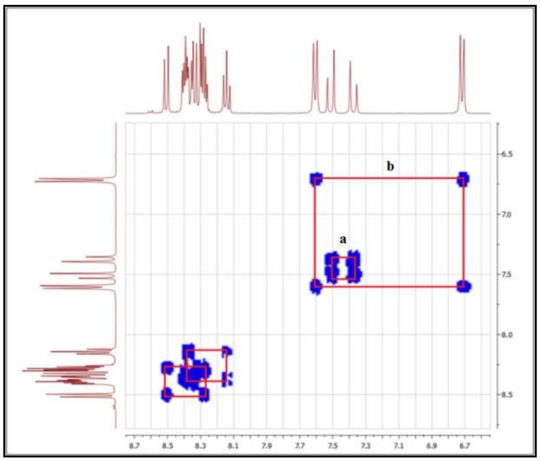

Figure 2. COSY spectrum of **3a**

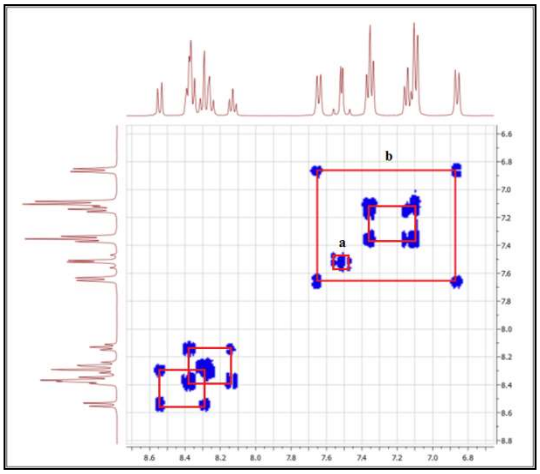

Figure 3. COSY spectrum of **3b**

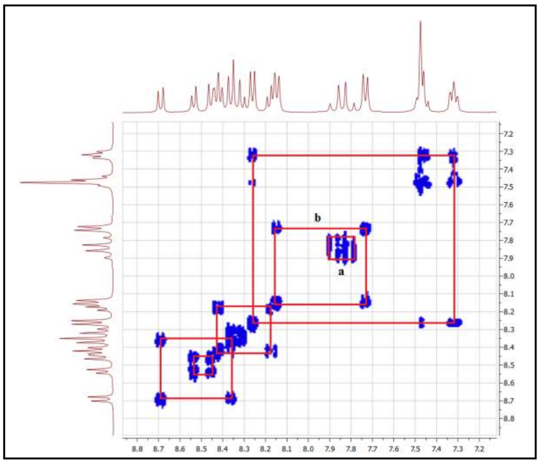

Figure 4. COSY spectrum of **3c**

At the same time, to show correlation between specific protons and associated chemical shifts, 2D-NMR technique (COSY) was applied to target compounds. In the first spectrum (Figure 2), rectangle a shows the couplings of α,β-unsaturated double bond protons of **3a** (doublets at $\delta = 7.35$ and 7.55). In addition, the coupling of dimethylamine attached phenyl group's hydrogens can be clearly seen in rectangle b (doublets at $\delta = 7.61$ and 6.71). Lastly, the correlations between pyrene protons can be roughly seen at lower fields in the spectrum.

Figure 3 shows the correlation spectrum of **3b** target compound and rectangle a visualizes the coupling of α,β-unsaturated double bond protons (doublets at $\delta = 7.54$ and 7.49). In addition, rectangle b also shows the diphenylamino group bonded phenyl ring on the proposed structure (doublets at $\delta = 7.64$ and 6.86). This spectrum also shows the pyrene related protons at lower fields as in Figure 2.

The last spectrum in Fig 4 is of the carbazole containing structure (**3c**). In the spectrum, olefinic bond and benzaldehyde related phenyl ring's protons are shown in rectangle a (doublets at $\delta = 7.88$ and 7.81) and b (doublets at $\delta = 8.15$ and 7.73), respectively. Moreover, the couplings of pyrene and carbazole protons can be separately seen at different chemical shifts.

3.3 *Absorption Studies*

Absorption spectra of **3a-c** were recorded in six common organic solvents at 10^{-5} M concentrations to understand the photophysical manners. All the absorption results for target compounds were summarized in Table 1 and the absorption spectra can be seen in Figure 5, Figure 6, and Figure 7, as well.

Table 1. Absorption maxima values of **3a-c** (nm)

	3a	**3b**	**3c**
Cyclohexane	402	412	394
Acetonitrile	418	418	384
Tetrahydrofuran	410	422	394
Dimethylformamide	420	424	392
Dichloromethane	414	426	392
Ethanol	428	428	396

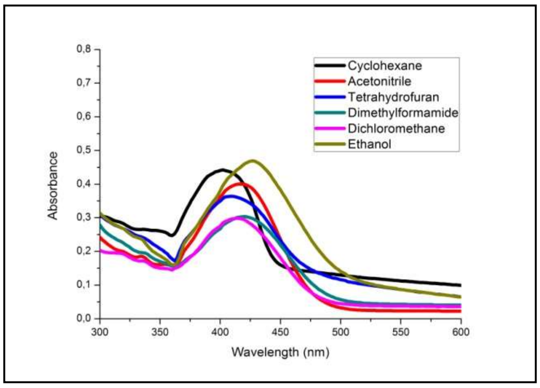

Figure 5. Absorption spectrum of **3a**

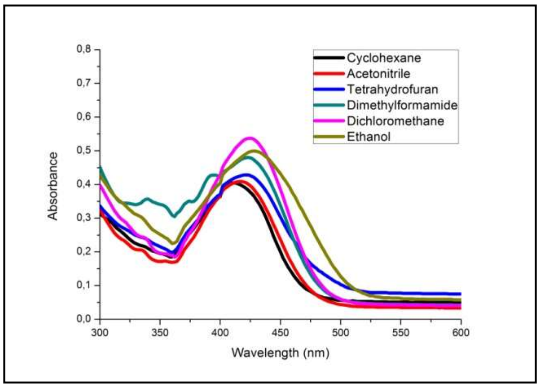

Figure 6. Absorption spectrum of **3b**

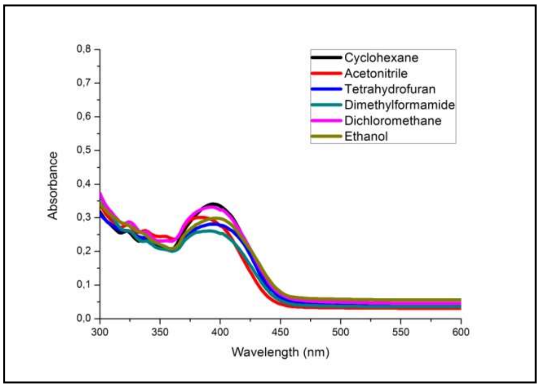

Figure 7. Absorption spectrum of **3c**

As shown in absorption spectra (Figure 5, Figure 6, Figure 7) and Table 1, target chalcone compounds exhibited one main absorption peak between ~380-430 nm for each studied solvents and these signals are assigned to the n-π* and π-π* transitions. These shapes are unstructured, and this may come from intramolecular charge transfers from electron donating groups to α,β-unsaturated carbonyl groups. By analyzing the results, a correlation was shown between λ_{abs} values and polarity of used solvents. In other words, these changings are affected from polarity and increasing polarity causes bathochromic shifts as expected. In addition, compound **3c** exhibited lower wavelength absorption when compared to others (**3a** and **3b**). This situation may come from higher electron donation ability of dimethylamino and diphenylamino groups.

3.4 *Thermal Analysis*

Thermal gravimetric analysis studies were carried out to understand the thermal perspective and obtained thermograms gave many important data about synthesized chalcone compounds (**3a-c**). As a main result, all three compounds showed good thermal stability (with max. 10 % weight loss) up to ~350 °C (Figure 6, Figure 7, and Figure 8). In a small range on this temperature, each compound exhibited different thermal behaviours with main weight losses in the range of 325-600, 350-450, and 350-450 for **3a**, **3b**, and **3c**, respectively. In Figure 8 and Figure 9, it can be seen slow degradations and weight losses while the weight loss is sharp in the thermogram of **3c** (Figure 10).

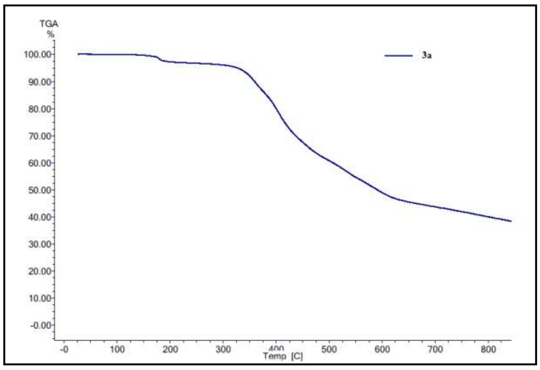

Figure 8. Thermogram of **3a**

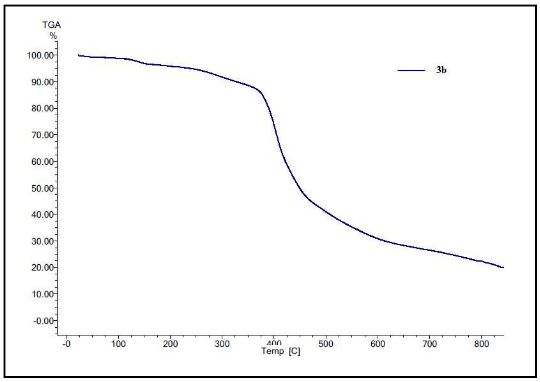

Figure 9. Thermogram of **3b**

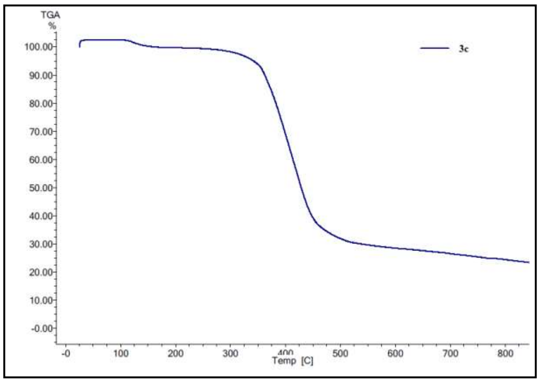

Figure 10. Thermogram of **3c**

3.5 *Surface Analysis*

To determine the possibility of using in organic electronics, the SEM images of all compounds (**3a-c**) were obtained by coating on ITO glass. A several solvent was used to generate thin film and dichlorobenzene was the most appropriate for the coating processes of these compounds. From all the images (Figure 11, Figure 12 and Figure 13), it can be understood that all the compounds have good ability to generate thin film (dark coloured area) and have potential to be used in organic electronics.

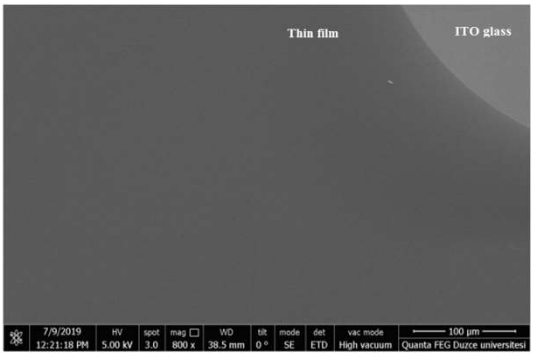

Figure 11. SEM image of **3a** thin film

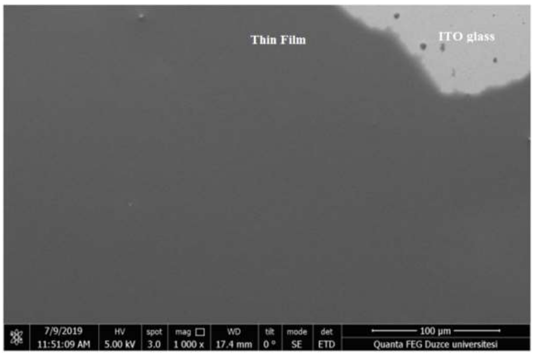

Figure 12. SEM image of **3b** thin film

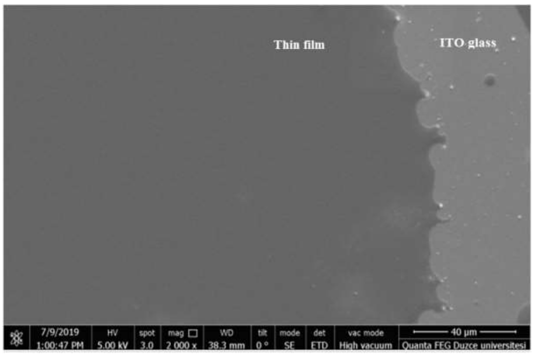

Figure 13. SEM image of **3c** thin film

4. CONCLUSIONS

Three chalcone compounds were successfully synthesized via condensation of 1-acetylpyrene with dimethylamine, diphenylamine or carbazole containing benzaldehyde derivatives at basic conditions. The yields were excellent in the range of 87-92%. In structural characterisation, it was shown trans configuration by consulting the coupling constants in ^{1}H NMR. UV-vis studies showed visible region absorption between ~380-430 nm. For each compound, λ_{max} values were quite different at different solvents and this was evidence of solvent type dependent absorption. The thermal studies showed that the target compounds were quite stable up to 350 °C. This thermal stability of the compounds can prevent from degradation and it ensures ultimately advantageous results at the end of application. Lastly, SEM images were taken to determine surface and film generation properties which are important for usability in organic electronics. The results were quite promising due to good film generation ability of each compound and these compounds might be used in organic electronics. By taking into account of electron donating and highly conjugated structures, absorption properties, high thermal stabilities, and good film generation properties, synthesized compounds can be promising candidates for organic electronics and dependent area.

ACKNOWLEDGMENTS

The author is thankful to Department of Chemistry in Faculty of Arts and Sciences of Düzce University and Prof. Dr. Sefa DURMUŞ for all kind support.

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