

Scientific paper

Synthesis, Characterization, and Investigation of Mesomorphic Properties of a New 2,5-Bis-(4-alkanoyloxybenzylidene)cyclopentan-1-one

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Abstract

A new set of cyclopentanone chalcone esters 2,5-bis-(4-alkanoyloxybenzylidene)cyclopentan-1-one (**B2–B10**) has been synthesized and monitored by TLC. Structures of these compounds were determined by spectroscopic techniques (FTIR, ¹H NMR, and mass spectrometry). Differential scanning calorimetry (DSC) and polarized optical microscopy were used to evaluate their transition temperatures and mesophase properties (POM) throughout heating and cooling scans. The thermal data indicate that the compounds **B5–B10** have mesomorphic properties with thermal stabilities; the data also reveal that the compounds **B6–B10** are monotropic, whereas **B5** is enantiotropic. **B6**, **B7**, and **B9** only have a nematic phase, but **B8** and **B10** have a smectic phase followed by a nematic phase, and **B5** only has a smectic phase. In addition, the study reveals that the inclusion of an acyl group as a terminal chain had the opposite effect on isotropization temperatures for compounds **B6**, **B8**, and **B10**, resulting in an increase in transition temperatures and a decrease in mesophase stability. The lack of a smectic phase in **B7** and **B9** compounds could be attributed to the narrow phase temperature range, which makes examination difficult, or to the molecules' lack of lateral attraction.

Keywords: Cyclopentanone chalcone, nematic, smectic, enantiotropic, monotropic.

1. Introduction

Chalcone is the common name for flavonoids, the name chalcone comes from the Greek word “chalcos”, which means “bronze”, because most natural chalcones have a bronze color.¹ Chalcones have two aromatic rings linked by an unsaturated α,β -ketone. Chemically, they are open-chain flavonoids with two aromatic rings bonded by a three-carbon atom (unsaturated carbonyl) system (Figure 1). In other words, “chalcones are structural derivatives of 1,3-diphenylprop-2-en-1-one”.^{2–4}

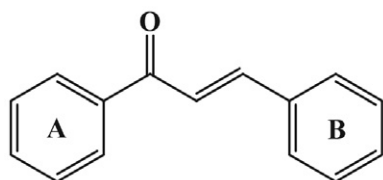


Figure 1: General structure of a chalcone

The Claisen–Schmidt condensation reaction is often used to prepare chalcones, which involves the condensation of aldehydes and ketones in the presence of a base or acid catalyst, followed by a dehydration process.⁵ Chalcones have recently received a lot of attention, not only from the synthetic and biosynthetic standpoint, but also because of their biological activities,⁶ which include anti-cancer,^{7–9} GSK-3 inhibition and antimicrobial activity,¹⁰ anti-HIV,¹¹ and antimalarial properties.¹² The target of this study is to synthesize cyclopentanone chalcone esters and investigate their liquid crystal properties and the influence of chain length on these properties.

2. Experimental Section

2.1. Materials

All chemicals were purchased from Sigma–Aldrich and were used without further purification. Melting points

were determined in an open capillary tube and are uncorrected. A Tensor 27 Bruker, Germany spectrometer was used to record infrared spectra as ATR (range 4000–600 cm^{-1}). The ^1H NMR spectra were recorded on a Bruker Ultraschield 400 MHz NMR spectrometer, Germany, using $\text{DMSO}-d_6$ as the solvent. The chemical shifts are reported as δ values (in ppm). Mass spectrum analyses were performed by the Agilent Technology MS 5973 device. POM equipped with a hot stage and Mettler Toledo DSC 823 (DSC) at a heating rate of $20\text{ }^\circ\text{C min}^{-1}$ was used for the investigation of phase transition temperatures.

2. 2. Synthesis of 2,5-Bis((*E*)-4-hydroxybenzylidene)cyclopentan-1-one (A)

In a 250 mL round-bottom flask containing 100 mL of ethanol, cyclopentanone (40 mmol) and 4-hydroxybenzaldehyde (80 mmol) were introduced and thoroughly mixed. Then, 40% sodium hydroxide solution (10 mL) was added slowly, and the reaction mixture was stirred overnight at room temperature. Thereafter, the reaction mixture was poured into a beaker containing crushed ice to quench the reaction and then neutralized with 10% HCl. The precipitate was filtered and recrystallized from absolute ethanol.

Green solid, yield 76%; mp $> 280\text{ }^\circ\text{C}$ (lit. $> 300\text{ }^\circ\text{C}$), $R_f = 0.8$ (acetone:hexane 5:5). IR (ATR) ν 3289 (O-H phenol), 3046 (C-H aromatic), 2973 (ν C-H_{al}), 1668 (ν C=O), 1561, 1509 (aromatic ring) cm^{-1} . ^1H NMR (400 MHz) δ 10.06 (lit. 10.06) (s, 2H, H₁, H_{1'}), 7.53 (d, 4H, H_{2,2'}, H_{2',2'}), 7.34 (lit. 7.34) (s, 2H, H₃, H_{3'}), 6.83–6.90 (d, 4H, H_{4,4'}, H_{4',4'}), 3.01 (lit. 3.01) (t, 4H, H_{5,5'}, H_{5',5'}).

2. 2. Synthesis of (1*E*,1'*E*)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene)bis(4,1-phenylene) dialkanoate B2–B10

To a 50 mL round-bottomed flask immersed in an ice bath and containing 20 mL of pyridine and compound A (1.7 mmol), an appropriate acid chloride (3.4 mmol) was added. The mixture was stirred at room temperature overnight. Thereafter, the mixture was poured into a beaker containing crushed ice, acidified with 10% HCl, and the solid product was filtered, washed with water, and recrystallized from absolute ethanol. TLC was performed using acetone-hexane (3:7) as the eluent solution.

2. 3. Characterization of Products B2–B10

((1*E*,1'*E*)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene))bis(4,1-phenylene) diacetate (B2)

Yellow solid, yield 77%; mp $200\text{--}202\text{ }^\circ\text{C}$ (decomp.), $R_f = 0.62$, IR (ATR) ν 3110 (C-H aromatic), 2981 (C-H_{al}), 1763 (ν C=O_{ester}), 1671 (ν C=O_{ketone}), 1594, 1505 (aro-

matic ring) cm^{-1} . ^1H NMR (400 MHz) δ 6.78–7.95 (d, 8H, Ar-H), 7.65 (s, 2H, CH=CH), 3.00 (t, 4H, saturated five membered ring), 2.40 (s, 6H, CH₃ groups).

((1*E*,1'*E*)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene))bis(4,1-phenylene) dipropionate (B3)

Yellow solid, yield 48%; mp $280\text{--}282\text{ }^\circ\text{C}$, $R_f = 0.81$, IR (ATR) ν 3114 (C-H aromatic), 2975 (C-H_{al}), 1759 (ν C=O_{ester}), 1675 (ν C=O_{ketone}), 1577, 1504 (aromatic ring), 752 (γ CH₂) cm^{-1} . ^1H NMR (400 MHz) δ 7.67–6.76 (d, 8H, Ar-H), 7.47 (s, 2H, CH=CH), 2.95–3.00 (t, 4H, saturated five membered ring), 1.25–2.52 (m, 10H, alkyl groups).

((1*E*,1'*E*)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene))bis(4,1-phenylene) dibutyrate (B4)

Yellow solid, yield 71%; mp $164\text{ }^\circ\text{C}$ (decomp.), $R_f = 0.76$, IR (ATR) ν 3123 (C-H aromatic), 2978 (C-H_{al}), 1754 (ν C=O_{ester}), 1672 (ν C=O_{ketone}), 1564, 1505 (aromatic ring), 743 (γ CH₂) cm^{-1} . ^1H NMR (400 MHz) δ 7.73–6.85 (d, 8H, Ar-H), 7.40 (s, 2H, CH=CH), 2.55–3.09 (t, 4H, saturated five membered ring), 0.89–2.50 (m, 14H, alkyl groups). MS m/z 434.3.

((1*E*,1'*E*)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene))bis(4,1-phenylene) dipentanoate (B5)

Deep yellow solid, yield 45%; mp $197\text{--}199\text{ }^\circ\text{C}$, $R_f = 0.72$, IR (ATR) ν 3119 (C-H aromatic), 2963 (C-H_{al}), 1752 (ν C=O_{ester}), 1687 (ν C=O_{ketone}), 1594, 1504 (aromatic ring), 754 (γ CH₂) cm^{-1} . ^1H NMR (400 MHz) δ 7.25–7.74 (d, 8H, Ar-H), 7.46 (s, 2H, CH=CH), 3.10 (t, 4H, saturated five membered ring), 0.95–2.50 (m, 18H, alkyl groups). MS m/z 460.7.

((1*E*,1'*E*)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene))bis(4,1-phenylene) dihexanoate (B6)

Yellow solid, yield 53%; mp $169\text{--}171\text{ }^\circ\text{C}$, $R_f = 0.74$, IR (ATR) ν 3057 (C-H aromatic), 2957 (C-H_{al}), 1753 (ν C=O_{ester}), 1688 (ν C=O_{ketone}), 1593, 1504 (aromatic ring), 736 (γ CH₂) cm^{-1} . ^1H NMR (400 MHz) δ 6.32–7.00 (d, 8H, Ar-H), 6.99 (s, 2H, CH=CH), 1.94–2.05 (t, 4H, saturated five membered ring), 0.30–1.11 (m, 22H, alkyl groups). MS m/z 490.4.

((1*E*,1'*E*)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene))bis(4,1-phenylene) diheptanoate (B7)

Brown solid, yield 28%; mp $170\text{--}172\text{ }^\circ\text{C}$, $R_f = 0.67$, IR (ATR) ν 3116 (C-H aromatic), 2987 (C-H_{al}), 1759 (ν C=O_{ester}), 1669 (ν C=O_{ketone}), 1597, 1509 (aromatic ring), 769 (γ CH₂) cm^{-1} . ^1H NMR (400 MHz) δ 6.88–7.98 (d, 8H, Ar-H), 7.51 (s, 2H, CH=CH), 2.46–3.43 (t, 4H, saturated five membered ring), 0.86–1.66 (m, 26H, alkyl groups).

((1*E*,1'*E*)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene))bis(4,1-phenylene) dioctanoate (B8)

Brown solid, yield 46%; mp $178\text{--}180\text{ }^\circ\text{C}$, $R_f = 0.71$, IR (ATR) ν 3080 (C-H aromatic), 2956 (C-H_{al}), 1748 (ν

$\text{C}=\text{O}_{\text{ester}}$, 1669 ($\nu \text{C}=\text{O}_{\text{ketone}}$), 1597, 1509 (aromatic ring), 748 (γCH_2) cm^{-1} . $^1\text{H NMR}$ (400 MHz) δ 6.90–7.98 (d, 8H, Ar-H), 7.46 (s, 2H, CH=CH), 2.78–3.30 (t, 4H, saturated five membered ring), 0.86–2.38 (m, 30H, alkyl groups).

((1E,1'E)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene))bis(4,1-phenylene) dinonanoate (B9)

Brown solid, yield 84%; mp 162–164 °C, $R_f = 0.71$, IR (ATR) ν 3057 (C-H aromatic), 2957 (C-H_{al}), 1752 ($\nu \text{C}=\text{O}_{\text{ester}}$), 1690 ($\nu \text{C}=\text{O}_{\text{ketone}}$), 1594, 1503 (aromatic ring), 747 (γCH_2) cm^{-1} . $^1\text{H NMR}$ (400 MHz) δ 6.88–7.75 (d, 8H, Ar-H), 7.40 (s, 2H, CH=CH), 2.50–3.35 (t, 4H, saturated five membered ring), 0.86–1.65 (m, 34H, alkyl groups).

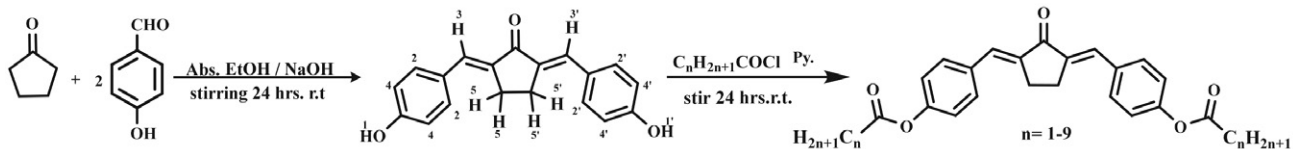
((1E,1'E)-(2-Oxocyclopentane-1,3-diylidene)bis(methaneylylidene))bis(4,1-phenylene) bis(decanoate) (B10)

Brown solid, yield 81%; mp 175–177 °C, $R_f = 0.61$, IR (ATR) ν 3101 (C-H aromatic), 2958 (C-H_{al}), 1752 ($\nu \text{C}=\text{O}_{\text{ester}}$), 1690 ($\nu \text{C}=\text{O}_{\text{ketone}}$), 1596, 1503 (aromatic ring), 741 (γCH_2) cm^{-1} . $^1\text{H NMR}$ (400 MHz) δ 6.89–7.73 (d, 8H, Ar-H), 7.32 (s, 2H, CH=CH), 2.52–3.07 (t, 4H, saturated five membered ring), 0.87–1.64 (m, 38H, alkyl groups). MS m/z 600.5.

3. Result and Discussion

3.1. Chemistry

Target ester compounds B2–B10 were synthesized using the method presented in scheme 1.



Scheme 1. Synthetic route for synthesis of compounds B2–B10

The presence of α -hydrogen atoms on both sides of the carbonyl group in the cyclopentanone molecule was used in the Claisen–Schmidt condensation reaction of cyclopentanone with 4-hydroxybenzaldehyde to produce two α,β -unsaturated groups of the chalcone compound 2,5-bis((E)-4-hydroxybenzylidene)cyclopentan-1-one (A). While esters B2–B10 were synthesized by reacting phenolic hydroxyl groups in chalcone A as a nucleophile with acid chlorides in the presence of pyridine as a solvent. This reaction was carried out in two steps. The nucleophile (OH) first attacks the carbonyl carbon atom of an acyl chloride, forming a tetrahedral intermediate that can eliminate the chloride as a leaving group.

3.2. Characterization

TLC was used to monitor the synthesis of chalcone A, with changes in product color and melting point serving

as a preliminary evidence.¹³ Thereafter, the FTIR spectrum (Figure 2) for compound A revealed a broad absorption band at 3289 cm^{-1} attributed to the O–H group, a medium absorption at 3046 cm^{-1} attributed to the aromatic C–H, a strong absorption band at 2973 cm^{-1} attributed to the C–H_{asy}(aliphatic) and the absorption at 2935 cm^{-1} that can be assigned to the C–H_{sy}(aliphatic), while the C=O group stretching vibration for ketone was observed at 1668 cm^{-1} .

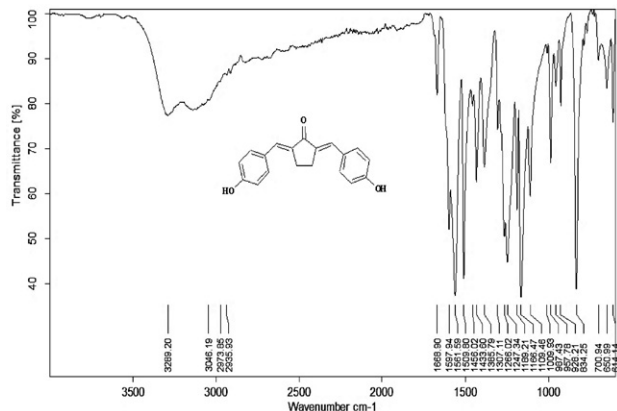


Figure 2: FT IR spectrum for chalcone A.

The $^1\text{H NMR}$ (Figure 3) spectrum of chalcone A reveals three distinct regions of chemical shifts. The signal of the hydroxyl proton appears as a singlet at δ 10.09 ppm, aromatic protons (8H) signals appear as two doublets in

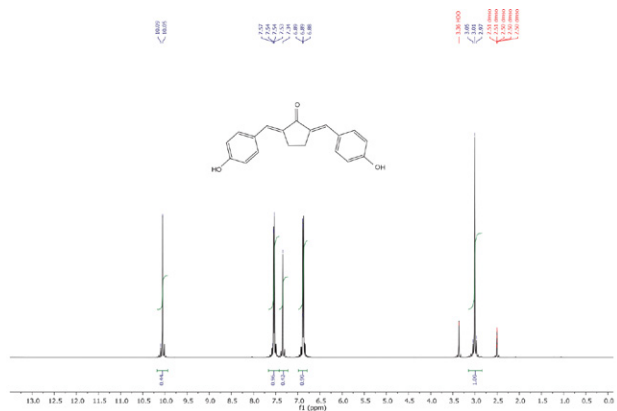


Figure 3: $^1\text{H NMR}$ spectrum for chalcone A.

the regions of δ 7.57–7.53 and 6.89–6.88 ppm, β -protons signal was observed as a singlet at δ 7.34 ppm, while a triplet signal at δ 3.01 ppm was ascribed to the two methylene groups in cyclopentane.

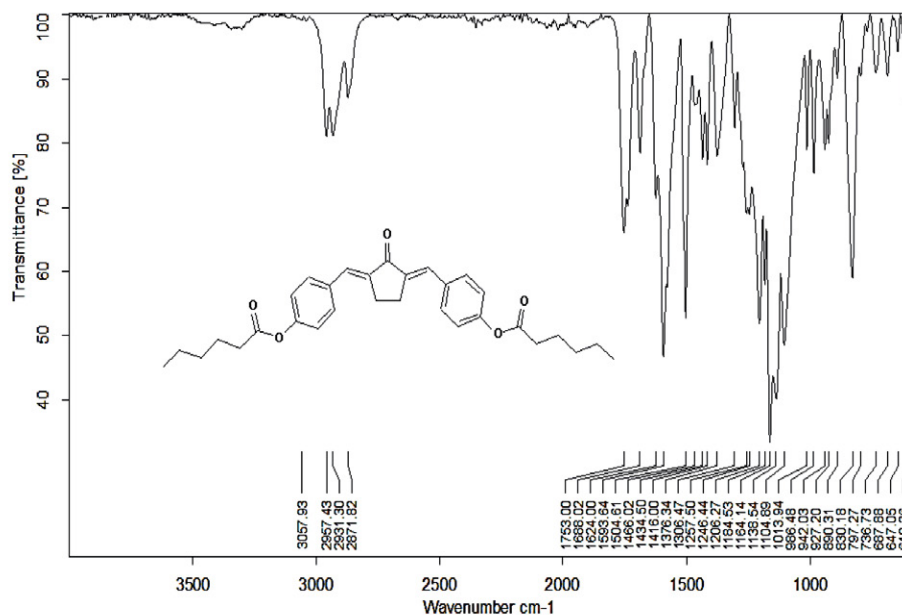


Figure 4: FT IR spectrum for chalcone **B6**.

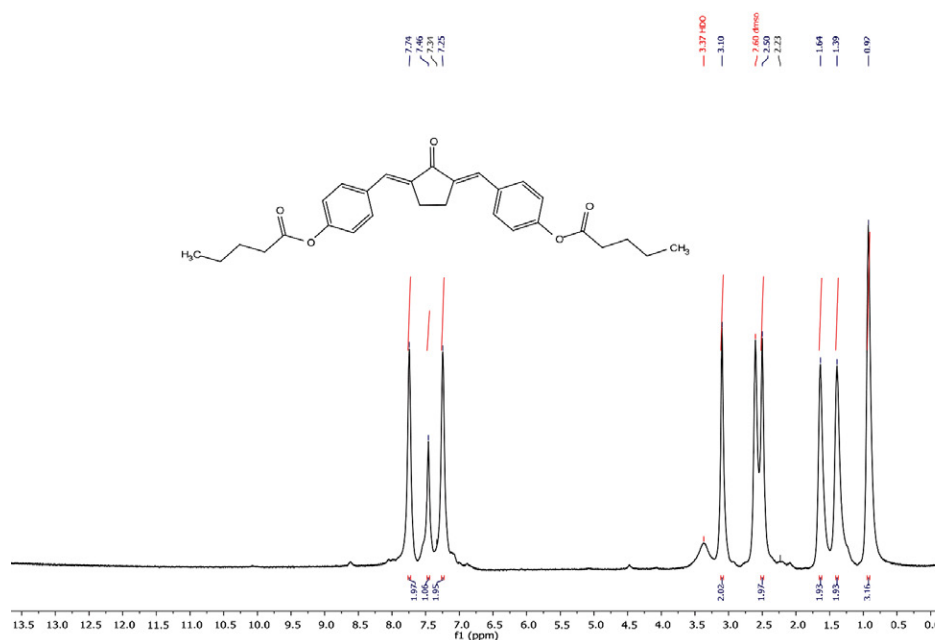


Figure 5: ^1H NMR spectrum for compound **B5**.

The FTIR spectra for compounds **B2–B10** (see Figure 4 for a representative case of **B6**) show the disappearance of the OH band for chalcone, and the appearance of an absorption band within the range of $3123\text{--}3110\text{ cm}^{-1}$ that is attributable to the aromatic C–H, a strong absorption band within the range of $1762\text{--}1748\text{ cm}^{-1}$ that is attributable to the C=O group of ester, and absorption band within the range of $1690\text{--}1669\text{ cm}^{-1}$ assigned to the C=O group for ketone.

According to ^1H NMR spectra for compounds **B2–B10**, the OH signal at 10.09 ppm attributable to compound

A has disappeared, aromatic proton signals were observed within the range δ 6.32–7.79 ppm, while proton signals for alkyl groups were observed within the range δ 0.25–3.23 ppm. See Figure 5 for a representative case of a ^1H NMR spectrum for compound **B5**.

3. 3. Mesomorphic Properties

The DSC and POM were utilized to investigate the characteristics of the phases of the 2,5-bis-(4-alkoxybenzylidene)cyclopentan-1-one compounds **B2–B10**

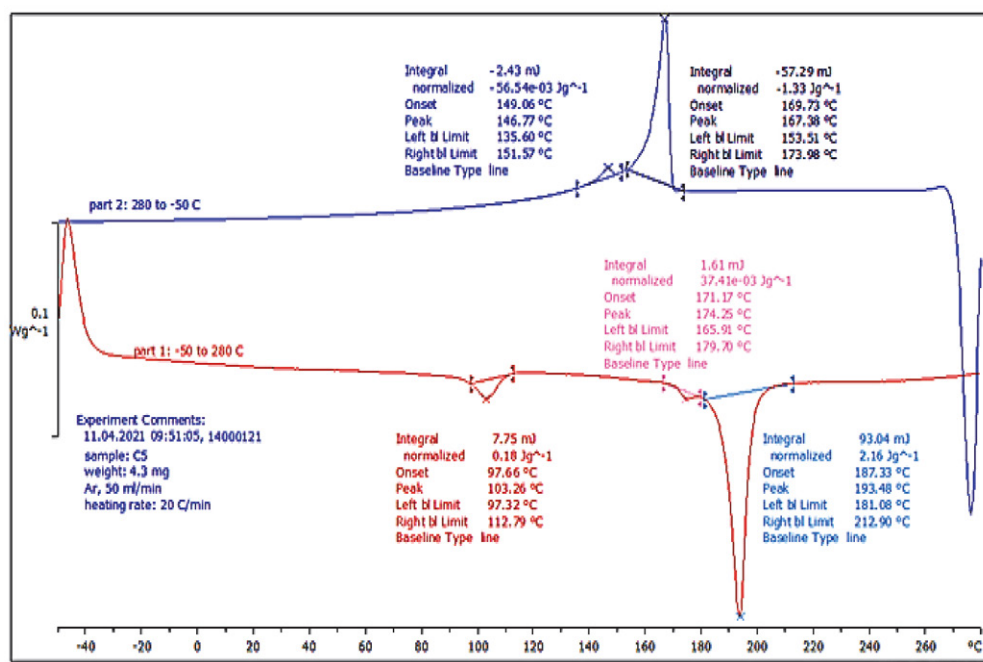


Figure 6: DSC of compound B5 during heating/cooling cycles

Table 1. Phase transitions and transition enthalpy for chalcone esters B5–B10 under heating and cooling cycle

Comp.	Transition Temperatures, °C (ΔH , kJ mol ⁻¹)	
	Heating	Cooling
B5	Cr 103.26 (0.0829) S _A 174.25 (17.22) S _C 193.48 (0.994) I Cr 70.99/0.0002 S _A 19.23/ 0.038 S _C -/ 0.002 I	I 167.38 (0.61) S _C 146.77 (26.0) Cr
B6	Cr 68.62 (3.058) N 168.70 (23.01) I Cr 100.08/0.008 N -/ 0.052 I	I 100.89 (10.26) Cr
B7	Cr 103.91 (3.39) N 164.10 (17.59) I Cr 60.19/ 0.009 N - / 0.040 I	I 142.23 (9.55) Cr
B8	Cr ₁ 87.88 (6.30) Cr ₂ 106.95 (1.08) S _A 134.28 (0.71) N 173.42 (16.94) I Cr ₁ 19.07/ 0.017 Cr ₂ 27.33/ 0.002 S _A 39.14 / 0.0004 N - / 0.037 I	I 147.54 (13.51) Cr
B9	Cr ₁ 122.92 (1.72) Cr ₂ 132.27 (0.36) N 166.99 (23.15) I Cr ₁ 9.35/0.0043 Cr ₂ 34.72/0.0009 N -/0.0526 I	I 150.77 (17.63) Cr
B10	Cr ₁ 21.01/0.0096 Cr ₂ 51.25/0.0019 S _A 32.78/0.018 S _C 8.84/0.0273 N -/0.0055 I	I 145.58 (10.51) Cr

upon heating and cooling, by tracing the thermal transitions in the DSC and confirming mesophases type by observing the texture using POM. Figure 6 shows the DSC thermogram for the compound B5 after a heating and cooling scan.

The transition temperatures, enthalpies, and entropies of the chalcone ester compounds B5–B10 from DSC are presented in Table 1.

The phase behavior investigation of chalcone ester compounds revealed that the compounds B5–B10 exhibit liquid crystal properties with thermal stabilities. Compounds B6–B10 were monotropic, while B5 was enantiotropic. Compounds B6, B7, and B9 have only a nematic phase, while B8 and B10 have a smectic phase followed by a

nematic phase, and B5 has only a smectic phase (Figure 7).

According to thermal degradation studies, compounds having odd methylene groups in the terminal alkoxy chain are more stable than those with even number of methylene groups.¹⁴ While the study of the data in Table 1 revealed that the introduction of an acyl group as a terminal chain had the opposite effect on isotropization temperatures for compounds B6, B8, and B10, increasing transition temperatures and decreasing their mesophase stability (Figure 6).

The lack of a smectic phase in compounds B7 and B9 could be due to the phase's narrow temperature range, which causes investigation difficulties, or to the molecules' lack of lateral attraction.

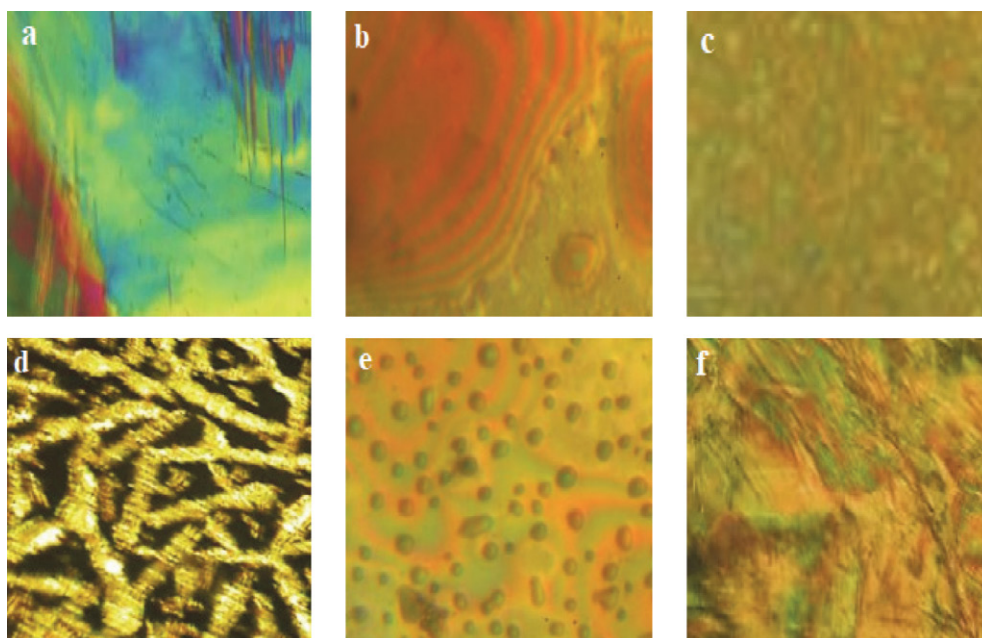


Figure 7: Polarized optical micrographs for: (a) compound B5 (SC at 174.25 °C); (b) compound B6 (N, at 68.62 °C); (c) compound B7 (N, mosaic at 103.91 °C); (d) compound B8 (SA, at 106.95 °C); (e) compound B9 (N, Schlieren, at 132.27 °C); (f) compound B10 (N, mosaic at 164.30 °C).

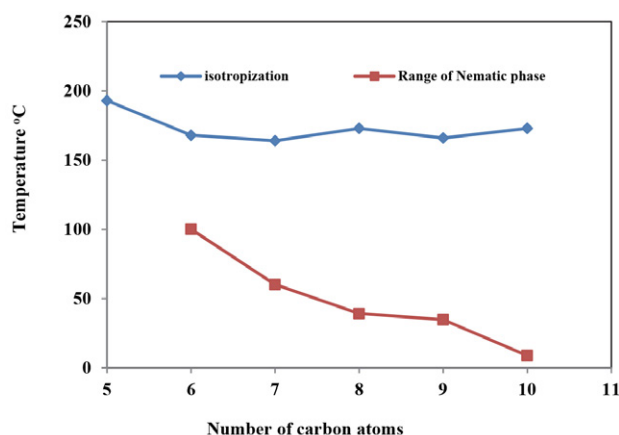


Figure 8: Relationship of isotropization and nematic phase range with the number of carbon atoms

4. Conclusion

The chalcone esters of cyclopentanone were successfully synthesized, and FTIR and ^1H NMR were used to characterize them. According to thermal data, compounds with 2–4 carbon atoms are non-mesogenic materials, while those with 5–10 carbon atoms are mesogenic materials. For compounds with 6, 8, and 10 carbon atoms, adding an acyl group as a terminal chain had the opposite impact on isotropization temperatures, causing an increase in transition temperatures and a decrease in mesophase stability.

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Povzetek

Sintetizirali smo novo serijo ciklopentanonskih halkonskih estrov 2,5-bis-(4-alkanoiloksibenziliden)ciklopentan-1-onov (**B2–B10**); reakcije smo nadzorovali s TLC. Strukture teh spojin smo določili s spektroskopskimi tehnikami (FTIR, ¹H NMR in masna spektrometrija). Diferenčna dinamična kalorimetrija (DSC) in polarizirana optična mikroskopija sta bili uporabljeni za določanje temperatur prehoda in mezofaznih lastnosti (POM) v celotnem območju segrevanja in ohlajanja vzorcev. Podatki termičnih analiz kažejo, da imajo spojine **B5–B10** mezomorfne lastnosti in da so termično stabilne; podatki tudi kažejo, da so spojine **B6–B10** monotropne, medtem ko je spojina **B5** enantiotropna. Spojine **B6**, **B7** in **B9** imajo samo nematsko fazo; pri spojinah **B8** in **B10** pa smektični fazi sledi nematska faza; spojina **B5** ima samo smektično fazo. Študije so tudi razkrile, da ima vključitev acilne skupine na terminalno mesto verige nasprotni učinek na izotropno temperaturo za spojine **B6**, **B8** in **B10**, kar povzroči povečanje temperatur prehoda in zmanjšanje mezofazne stabilnosti. Dejstvo, da spojin **B7** in **B9** nimata smektične faze, lahko pripišemo ozkim temperaturnim intervalom faznih prehodov, kar povzroča eksperimentalne težave, ali pa pomanjkanju lateralnih privlačnih sil med molekulami v teh dveh primerih.



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