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Combinatorial synthesis of isoxazole library and their liquid crystalline properties

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Abstract—Six types of isoxazoles including 60 molecules were synthesized using combinatorial synthesis on solid support in a parallel fashion. Highly regioselective 1,3-dipolar cycloaddition of the nitrile oxides to the triple bond of the solid-attached ethynylbenzamides gave isoxazoles on solid support in good yield. Thirty molecules of them exhibited mesomorphic properties. The combination of molecular mechanics calculations and X-ray diffraction experiments of **2g** and **3g** revealed that the smectic phase of **2g** had a bilayer structure, whereas **3g** showed a monolayer liquid crystalline phase. These are supported in comparison with the single crystal structures of **2g** and **3g**.

1. Introduction

Liquid crystals are partially ordered, anisotropic fluids, thermodynamically located between the solid state and the isotropic liquid. Liquid crystalline phases represent intriguing states of soft matter, combining order and mobility on a molecular level.[1] These unique phases create fascinating systems, which respond to external (magnetic, electronic, chemical, or mechanical) stimuli by finding a new configuration.[2] Thus, liquid crystalline materials have had great impact on recent development of mobile information technologies with numerous applications in optoelectric devices.

Classical rod-like liquid crystalline molecules are composed of anisometric units and flexible alkyl chains. Phenyl, biphenyl, terphenyl, cyclohexyl, etc., are often incorporated as an anisometric core. In recent years, many unique mesogens have been reported. Troponoid, expanded poly aromatic, metal-containing aromatic, fullerene-containing, self-assembling, and hydrogen-bonded mesogens[3] have been developed with their unique functions. Among them, bent-shaped (banana-shaped) liquid crystalline molecules show unique chiral mesogenic phases.[4] 3,5-Disubstituted isoxazole derivatives[5] formally belong to the class of the bent-shaped molecules; however, they exhibit classical nematic and smectic mesophases. Structure–property relationships in isoxazole liquid crystals still remain fairly uncharted. In particular, unsymmetrical diphenyl isoxazoles bearing an alkoxy chain at one end of the molecules and a polar group at the other end were limited.[6]

Combinatorial chemistry is one of the most promising tools for searching the desired functional compounds. Recently, liquid crystalline libraries of troponoid[7] and benzenoid amides[8] were prepared on solid support. In our previous report, we have systematically investigated the substitution effect on mesomorphic behaviors of anisometric 3,5-diphenyl isoxazole-based compounds having a cyano group using combinatorial chemistry.[9] In this paper, we report the extensive investigation of liquid crystalline properties for combinatorial library of 3,5-disubstituted isoxazoles.

1,3-Dipolar cycloaddition of nitrile oxide to triple bond is one of the most potential synthetic ways for the construction of an isoxazole core. In recent years, a number of solid-phase synthesis of isoxazoles via 1,3-dipolar cycloaddition have been reported.[10] We applied this method to the combinatorial synthesis of the isoxazole library using polymer supported phenylacetylene. Our strategy for the synthesis of the isoxazole liquid crystals on the solid support is outlined in Figure 1.

Phenylacetylene unit is immobilized on a polymer support by the amide linkage, which can be easily cleaved by acid catalysis and simultaneously converted to cyano group. The cycloaddition of nitrile oxides on solid support having a variety of alkoxy substituents provides isoxazoles **1** and **2** after removal from the solid support. Isoxazoles **3**–**6** are prepared by benzoylation of the resin-bound isoxazoles having a hydroxyl group, followed by cleavage from solid

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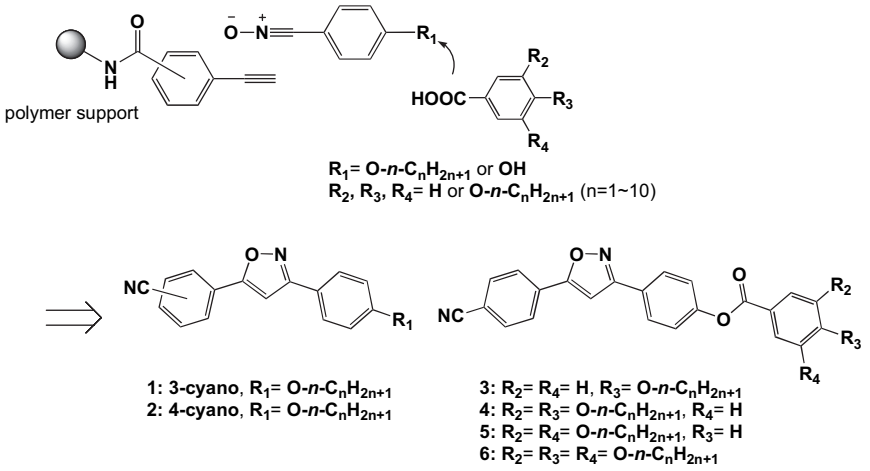


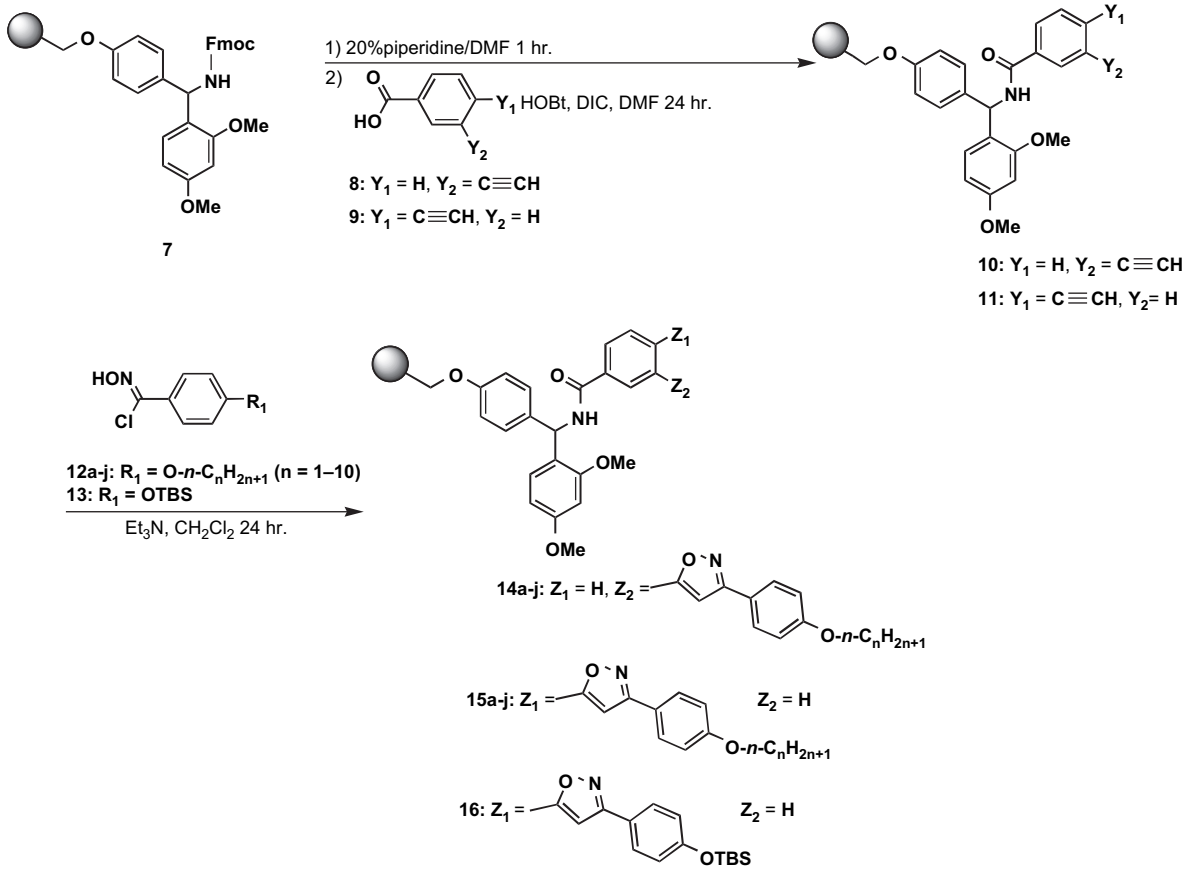
Figure 1. Synthetic strategy of isoxazole liquid crystal on solid support.

support. Isoxazole libraries **1**–**6** can allow us to carry out the systematic evaluation of the substitution effect on mesomorphism.

2. Results and discussion

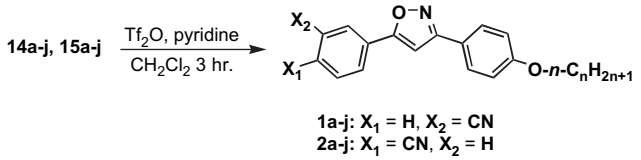
2.1. Library synthesis

Parallel synthesis of the isoxazole library on solid support was carried out using parallel synthesizer MiniBlock. Rink resin **7** was selected as a solid support. After the removal of the Fmoc group by treatment with piperidine in DMF, the condensation reaction of ethynylbenzoic acid **8** or **9** with *N*,*N*′-diisopropylcarbodiimide (DIC) and 1-hydroxybenzotriazole (HOBt) gave the corresponding products **10** and **11**. Resins **10** and **11** smoothly reacted with the nitrile oxides, generated in situ upon treatment of hydroximinoyl chlorides **12a**–**j**[11] and **13** with triethylamine, to provide the resin-bound diphenyl isoxazole **14a**–**j**, **15a**–**j**, and **16** (Scheme 1). Completion of the cycloaddition reactions was monitored by the disappearance of acetylene C–H band that appeared around 3286 cm^{-1} using FTIR spectroscopy. Trifluoromethanesulfonic anhydride worked nicely



Scheme 1.

to cleave the linkage to liberate the desired isoxazoles **1a–j** or **2a–j** into the solution (Scheme 2). After quick purification of the crude products by column chromatography, the isoxazoles were furnished in good yield (Table 1).



Scheme 2.

The *tert*-butyldimethylsilyl (TBS) group of **16** was removed upon treatment with tetrabutylammonium fluoride. The condensation reaction of benzoic acid derivatives **17a–j**, **18a–j**, **19a–j**, and **20a–j** with *N*,*N*′-diisopropylcarbodiimide in the presence of *N*,*N*-dimethylaminopyridine gave support-bound isoxazoles **21a–j**, **22a–j**, **23a–j**, and **24a–j**. Liberation of isoxazoles from the solid support upon treatment with trifluoromethanesulfonic anhydride gave the desired isoxazoles **3a–j**, **4a–j**, **5 a–j**, and **6a–j** (Scheme 3). All library members **1a–6j** were characterized by ^{1}H and ^{13}C NMR spectroscopies, high-resolution mass spectrometry, and elemental analysis.

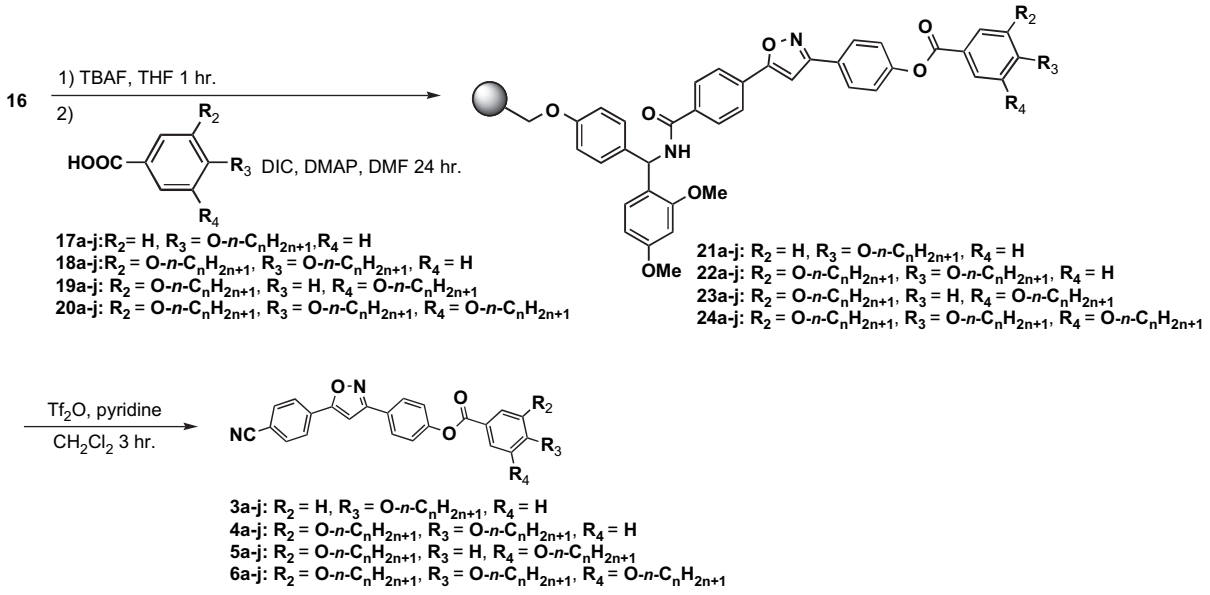
2.2. Mesomorphic behavior

The phase transition temperatures and the thermal behavior of compounds **1a–6j** were investigated by polarized optical microscopy and differential scanning calorimetric (DSC) measurements. The results are summarized in Tables 1 and 2. Mesomorphic behaviors were found in compounds **2**, whereas compounds **1** were not mesomorphic. This should be due to the larger molecular dipole of compounds **2** produced by the presence of the cyano group at *para*-position. The CN group of compounds **1** made molecular breadth wider to decrease the thermal stability of mesomorphic phases. Compounds **2** showed nematic (N) and smectic A (SmA) phases; the N phase was characterized by the observation of schlieren textures, and the SmA phase was assigned by the observation of fan textures. Shorter chained compounds **2a–f** displayed an N phase, whereas a SmA phase was observed in longer chained compounds **2i** and

Table 1. Isolated yields and transition temperatures of isoxazoles **1** and **2**[a]

	R_1	Yield (%)	Transition temp (°C)		R_1	Yield (%)	Transition temp (°C)
1a	OCH_3	72	Cr 159 Iso	**2a**	OCH_3	90	Cr 164 (12.1) N 223 (0.3) Iso
1b	OC_2H_5	85	Cr 157 Iso	**2b**	OC_2H_5	83	Cr 128 (26.8) N 199 (0.3) Iso
1c	OC_3H_7	49	Cr 138 Iso	**2c**	OC_3H_7	82	Cr 134 (26.4) [SmA 120[b]] N 178 (0.2) Iso
1d	OC_4H_9	64	Cr 131 Iso	**2d**	OC_4H_9	66	Cr 126 (27.8) N 185 (0.4) Iso
1e	OC_5H_{11}	77	Cr 116 Iso	**2e**	OC_5H_{11}	83	Cr 116 (29.2) N 180[b] Iso
1f	OC_6H_{13}	61	Cr 112 Iso	**2f**	OC_6H_{13}	69	Cr 112 (38.6) N 178 (0.6) Iso
1g	OC_7H_{15}	88	Cr 109 Iso	**2g**	OC_7H_{15}	67	Cr 113 (35.0) SmA 159 (0.1) N 178 (0.4) Iso
1h	OC_8H_{17}	62	Cr 110 Iso	**2h**	OC_8H_{17}	58	Cr 112 (31.4) SmA 168 (0.1) N 177 (0.1) Iso
1i	OC_9H_{19}	71	Cr 114 Iso	**2i**	OC_9H_{19}	74	Cr 92 (42.5) SmA 176 (2.5) Iso
1j	$OC_{10}H_{21}$	65	Cr 112 Iso	**2j**[c]	$OC_{10}H_{21}$	68	Cr 93 (29.5) SmA 173 (3.4) Iso

[a] Cr, crystals; N, a nematic phase; SmA, a smectic A phase; Iso, isotropic liquid. The transition temperatures and enthalpy changes (kJ/mol) shown in parentheses were obtained during the first heating process. [] means monotropic transition temperatures, which were obtained by cooling.
[b] Data could not be determined from DSC.
[c] A mixture of compound **2j** and its regioisomer was reported.[6]



Scheme 3.

Table 2. Isolated yields and transition temperatures of isoxazoles **3**–**6**[a]

	R_2	R_3	R_4	Yield (%)	Transition temp (°C)
3a	H	OCH_3	H	99	Cr 213 (36.3) N 352 Dec
3b	H	OC_2H_5	H	99	Cr 187 (42.9) N 366 Dec
3c	H	OC_3H_7	H	99	Cr 154 (28.1) SmA 221[b] N 355 Dec
3d	H	OC_4H_9	H	99	Cr 159 (33.0) SmA 262[b] N 356 Dec
3e	H	OC_5H_{11}	H	99	Cr 138 (25.8) SmA 278[b] N 340 Dec
3f	H	OC_6H_{13}	H	86	Cr 130 (57.2) SmA 300[b] N 328 Dec
3g	H	OC_7H_{15}	H	99	Cr 121 (27.4) SmA 291[b] N 304 Dec
3h	H	OC_8H_{17}	H	84	Cr 116 (27.7) SmA 310[b] N 313 Dec
3i	H	OC_9H_{19}	H	99	Cr 121 (49.2) SmA 318 Dec
3j	H	$OC_{10}H_{21}$	H	44	Cr 118 (41.5) SmA 300 Dec
4a	OCH_3	OCH_3	H	68	Cr 225 (45.6) N 284 (0.2) Iso
4b	OC_2H_5	OC_2H_5	H	92	Cr 189 (43.5) SmA 193[b] N 250 (0.6) Iso
4c	OC_3H_7	OC_3H_7	H	99	Cr 182 (35.6) SmA 222 (1.2) N 228 (0.4) Iso
4d	OC_4H_9	OC_4H_9	H	80	Cr 172 (34.9) SmA 220 (4.0) Iso
4e	OC_5H_{11}	OC_5H_{11}	H	48	Cr 159 (35.2) SmA 215 (4.9) Iso
4f	OC_6H_{13}	OC_6H_{13}	H	66	Cr 156 (33.9) SmA 205 (2.2) Iso
4g	OC_7H_{15}	OC_7H_{15}	H	82	Cr 152 (32.9) SmA 206 (2.3) Iso
4h	OC_8H_{17}	OC_8H_{17}	H	82	Cr 151 (39.0) SmC 157[b] SmA 203 (4.0) Iso
4i	OC_9H_{19}	OC_9H_{19}	H	71	Cr 147 (39.7) SmC 153[b] SmA 192 (3.6) Iso
4j	$OC_{10}H_{21}$	$OC_{10}H_{21}$	H	99	Cr 145 (41.0) SmC 159[b] SmA 188 (3.7) Iso
5a	OCH_3	H	OCH_3	99	Cr 223 Iso
5b	OC_2H_5	H	OC_2H_5	99	Cr 194 Iso
5c	OC_3H_7	H	OC_3H_7	31	Cr 175 Iso
5d	OC_4H_9	H	OC_4H_9	56	Cr 152 Iso
5e	OC_5H_{11}	H	OC_5H_{11}	99	Cr 144 Iso
5f	OC_6H_{13}	H	OC_6H_{13}	52	Cr 137 Iso
5g	OC_7H_{15}	H	OC_7H_{15}	34	Cr 135 Iso
5h	OC_8H_{17}	H	OC_8H_{17}	36	Cr 141 Iso
5i	OC_9H_{19}	H	OC_9H_{19}	71	Cr 128 Iso
5j	$OC_{10}H_{21}$	H	$OC_{10}H_{21}$	97	Cr 133 Iso
6a	OCH_3	OCH_3	OCH_3	84	Cr 214 Iso
6b	OC_2H_5	OC_2H_5	OC_2H_5	99	Cr 192 Iso
6c	OC_3H_7	OC_3H_7	OC_3H_7	89	Cr 188 Iso
6d	OC_4H_9	OC_4H_9	OC_4H_9	39	Cr 139 Iso
6e	OC_5H_{11}	OC_5H_{11}	OC_5H_{11}	79	Cr 117 Iso
6f	OC_6H_{13}	OC_6H_{13}	OC_6H_{13}	62	Cr 102 Iso
6g	OC_7H_{15}	OC_7H_{15}	OC_7H_{15}	55	Cr 102 Iso
6h	OC_8H_{17}	OC_8H_{17}	OC_8H_{17}	46	Cr 109 Iso
6i	OC_9H_{19}	OC_9H_{19}	OC_9H_{19}	84	Cr 112 Iso
6j	$OC_{10}H_{21}$	$OC_{10}H_{21}$	$OC_{10}H_{21}$	89	Cr 107 Iso

[a] The transition temperatures and enthalpy changes (kJ/mol) shown in parentheses were obtained during the first heating process. The transition temperatures of **5** and **6** were determined by the microscopic observation SmC, a smectic C phase; Dec, decomposition.
[b] Data could not be determined from DSC.

2j. Extension of the alkyl chain of **2** produced a SmA phase in which the molecules are more ordered than in an N phase. These trends suggest that the extension of the flexible alkyl chains creates well ordered mesomorphic phases.

Compounds **3** and **4** exhibit smectic and nematic phases. The additional benzene rings connected to the ester linkage increased the thermal stability of the liquid crystalline phases (**2** vs **3**), indicating that the segregation between the aromatic units and the flexible chains is well established in the liquid crystalline phases of **3**. The mesophases of **4** are thermally less stable than those of **3**. The presence of the more alkyl chains at *meta*-positions on the benzene ring increases the volumes of the flexible chains due to their dynamic movement; this should disrupt the order of the anisometric layers to make it less stable. Smectic C (SmC) phases were observed only in compounds **4h**–**j**, determined from the observation of broken-fan and schlieren textures. These compounds have the two long alkyl chains at *meta*- and *para*-position, bringing the slightly tilted arrays of the liquid crystalline phases. Compounds **5** and **6** are not mesomorphic; their bulky chains should interrupt the segregation of their anisometric units.

2.3. X-ray diffraction study

In order to obtain the structural information of molecular array in the liquid crystalline phases, the X-ray diffraction measurements (XRD) of **2g**, **3d**, **3g**, **4g**, and **4i** were carried out in the smectic phases. The smectic phase of **2g** showed the layer spacing *d* value (30.8 Å) larger than the calculated molecular length of **2g** (20 Å), indicating that the molecules should interdigitate and form a bilayer structure in the smectic A phase. In contrast, the layer spacing *d* values of smectic phases for **3d**–**4i** are consistent with the calculated molecular lengths (Table 3). This suggested that these molecules create simple monolayer structures in the smectic phases.

2.4. Crystal structures

Crystal structures of **2g**[9] and **3g**[13] gave an insight into the structures of the molecular arrays in the liquid crystalline phases. In the X-ray crystallography of **2g**, the rigid diphenyl isoxazole cores nicely stack in the antiparallel orientation due to the strong dipole–dipole interaction between the cyano groups (Fig. 2). This antiparallel orientation

Table 3. Layer spacing *d* values and calculated molecular lengths of **2**, **3**, and **4**

Compd	R_1	*d* (Å)	Temp (°C)[b]	Molecular lengths (Å)[c]
2g	OC_7H_{15}	30.8 (SmA)[a]	140	23.1
3d	OC_4H_9	23.1 (SmA)[a]	240	25.4
3g	OC_7H_{15}	28.1 (SmA)[a]	280	28.5
4g	OC_7H_{15}	30.9 (SmA)[a]	200	28.4
4i	OC_9H_{19}	31.0 (SmC)[a]	152	30.9
		32.1 (SmA)[a]	180	

[a] The mesogenic phases are indicated.
[b] The XRD were measured at the temperatures.
[c] The molecular lengths were calculated by MacroModel V. 6.5 using amber* force field.[12]

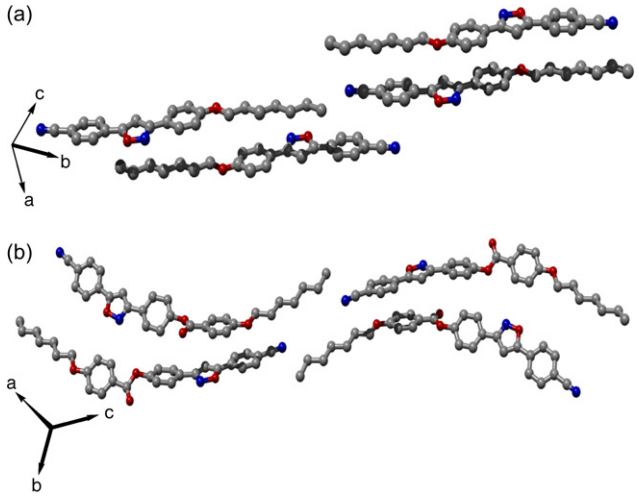


Figure 2. Packing of molecules (a) **2g** and (b) **3g**.

rationalizes that the bilayer structure of **2g** forms in the SmA phase.

In contrast, the crystal structure of **3g** disclosed that the cyano groups stay away from each other in the antiparallel orientation; the dipole–dipole interaction of the cyano groups is too weak to provide the bilayer structure. As a result, the mesogens having 4-alkoxybenzene carbonyloxy groups form the monolayer structures in their smectic phases.

3. Conclusion

We constructed 60 members of the isoxazole library using combinatorial synthesis on solid support. The isoxazoles **2** and **3** with alkyl chains showed SmA and N phases while the isoxazoles **4** with two alkyl chains presented SmC phases in addition to SmA and N phases. Combinatorial synthesis of liquid crystalline compounds has been shown to be potential for the systematic investigation for their functions, resulting from characteristics of their structures.

4. Experimental

4.1. General

^{1}H and ^{13}C NMR spectra were measured with a Varian Mercury 300 or JEOL-Lambda 500 spectrometer using a residual solvent signal as internal standard. All NMR spectra were recorded in $CDCl_3$ unless otherwise indicated. Mass spectra were recorded with a JEOL JMX-SX 102 mass spectrometer. IR spectra were measured on a JASCO FT/IR-420S spectrometer. Elemental analyses were performed on a Perkin–Elmer 2400CHN elemental analyzer. The mesomorphic phase and the transition temperatures were observed by a polarizing microscope Olympus (BHSP BH-2) equipped with a hot stage (Linkam TH-600RMS). The enthalpy changes were measured on a Shimadzu DSC-50 differential scanning calorimeter. The X-ray diffraction measurements were carried out with a Rigaku Rint 2100 system using Ni-filtered Cu Kα radiation at various temperatures. The measuring temperatures were controlled with a Linkam HFS-91 hot stage. The X-ray diffraction analysis was performed with Mac Science DIP2030 or Bruker SMART-APEX at ambient temperature.

All reactions were carried out under an argon atmosphere unless otherwise noted. THF was freshly distilled from Na–benzophenone. Dichloromethane (DCM) and dimethylformamide (DMF) were freshly distilled from CaH_2. Column chromatography was performed using Merck silica gel (70–230 mesh).

4.2. Preparations of benzohydroximoyl chloride 12a–j

4.2.1. 4-Methoxybenzohydroximoyl chloride 12a. See Ref. 14.

4.2.2. 4-Ethoxybenzohydroximoyl chloride 12b. See Ref. 15.

4.2.3. 4-Propoxybenzohydroximoyl chloride 12c. To a solution of 4-propyloxybenzaldoxime[16] (400 mg, 2.2 mmol) in DMF (2 ml) at 0 °C was added *N*-chlorosuccinimide (300 mg, 2.25 mmol). After stirred at room temperature for 1 h, the reaction mixture was poured into four volumes of water and extracted with ether. The organic layer was washed three times with water, dried over Na_2SO_4, and concentrated under vacuum to give the product (430 mg, 91%). The product prepared in this manner did not require further purification for conversion to the isoxazoles. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, 2H, J=9.0 Hz), 6.90 (d, 2H, J=9.0 Hz), 3.95 (t, 2H, J=6.6 Hz), 1.77–1.88 (m, 2H), 1.04 (t, 3H, J=7.5 Hz).

4.2.4. 4-Butoxybenzohydroximoyl chloride 12d. See Ref. 17.

4.2.5. 4-Pentyloxybenzohydroximoyl chloride 12e. To a solution of 4-pentyloxybenzaldoxime[16] (200 mg, 0.96 mmol) in DMF (2 ml) at 0 °C was added *N*-chlorosuccinimide (133 mg, 1.0 mmol). After stirred at room temperature for 1 h, the reaction mixture was poured into four volumes of water and extracted with ether. The organic layer was washed three times with water, dried over Na_2SO_4, and concentrated under vacuum to give the product (209 mg, 96%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, 2H, J=9.0 Hz), 6.90 (d, 2H, J=9.0 Hz), 3.99 (t, 2H, J=6.6 Hz), 1.75–1.85 (m, 2H), 1.35–1.49 (m, 4H), 0.93 (t, 3H, J=7.2 Hz).

4.2.6. 4-Hexyloxybenzohydroximoyl chloride 12f. To a solution of 4-hexyloxybenzaldoxime[16] (400 mg, 1.8 mmol) in DMF (2 ml) at 0 °C was added *N*-chlorosuccinimide (240 mg, 1.8 mmol). After stirred at room temperature for 1 h, the reaction mixture was poured into four volumes of water and extracted with ether. The organic layer was washed three times with water, dried over Na_2SO_4, and concentrated under vacuum to give the product (439 mg, 90%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, 2H, J=8.7 Hz), 6.90 (d, 2H, J=8.7 Hz), 3.99 (t, 2H, J=6.6 Hz), 1.74–1.84 (m, 2H), 1.33–1.48 (m, 6H), 0.91 (t, 3H, J=6.9 Hz).

4.2.7. 4-Heptyloxybenzohydroximoyl chloride 12g. To a solution of 4-heptyloxybenzaldoxime[16] (1 g, 5.17 mmol) in DMF (5 ml) at 0 °C was added *N*-chlorosuccinimide (725 mg, 5.43 mmol). After stirred at room temperature for 1 h, the reaction mixture was poured into four volumes of water and extracted with ether. The organic layer was washed three times with water, dried over Na_2SO_4, and concentrated under vacuum to give the product (1.28 g, 92%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, 2H, J=9.0 Hz), 6.90 (d, 2H, J=9.0 Hz), 3.99 (t, 2H, J=6.6 Hz), 1.75–1.84 (m, 2H), 1.31–1.45 (m, 8H), 0.89 (t, 3H, J=6.9 Hz).

4.2.8. 4-Octyloxybenzohydroximoyl chloride 12h. To a solution of 4-octyloxybenzaldoxime[16] (450 mg, 1.8 mmol) in DMF (2 ml) at 0 °C was added *N*-chlorosuccinimide (240 mg, 1.8 mmol). After stirred at room temperature for 1 h, the reaction mixture was poured into four volumes of water and extracted with ether. The organic layer was washed three times with water, dried over Na_2SO_4, and concentrated under vacuum to give the product (450 mg, 88%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, 2H, J=8.7 Hz), 6.90 (d, 2H, J=8.7 Hz), 3.98 (t, 2H, J=6.6 Hz), 1.74–1.84 (m, 2H), 1.29–1.47 (m, 10H), 0.88 (t, 3H, J=6.6 Hz).

4.2.9. 4-Nonyloxybenzohydroximoyl chloride 12i. To a solution of 4-pentyloxybenzaldoxime[16] (200 mg, 0.76 mmol) in DMF (2 ml) at 0 °C was added *N*-chlorosuccinimide (152 mg, 1.1 mmol). After stirred at room temperature for 1 h, the reaction mixture was poured into four volumes of water and extracted with ether. The organic layer was washed three times with water, dried over Na_2SO_4, and concentrated under vacuum to give the product (225 mg, 99%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, 2H, J=8.7 Hz), 6.90 (d, 2H, J=8.7 Hz), 3.98 (t, 2H, J=6.6 Hz), 1.77–1.81 (m, 2H), 1.28–1.46 (m, 12H), 0.88 (t, 3H, J=6.9 Hz).

4.2.10. 4-Decyloxybenzohydroximoyl chloride 12j. To a solution of 4-decyloxybenzaldoxime[16] (550 mg, 2 mmol) in DMF (2 ml) at 0 °C was added *N*-chlorosuccinimide (280 mg, 1.0 mmol). After stirred at room temperature for 1 h, the reaction mixture was poured into four volumes of water and extracted with ether. The organic layer was washed three times with water, dried over Na_2SO_4, and concentrated under vacuum to give the product (465 mg, 75%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, 2H, J=9.0 Hz), 6.90 (d, 2H, J=9.0 Hz), 3.98 (t, 2H, J=6.6 Hz), 1.74–1.84 (m, 2H), 1.27–1.51 (m, 14H), 0.88 (t, 3H, J=6.6 Hz).

4.2.11. 4-[(*tert*-Butyldimethylsilyl)oxy]benzohydroximoyl chloride 13. To a solution of 4-[(*tert*-butyldimethylsilyl)oxy]benzaldoxime[18] (800 mg, 3.2 mmol) in DMF (4 ml) at 0 °C was added *N*-chlorosuccinimide (446 mg, 3.3 mmol). After stirred at room temperature for 1 h, the reaction mixture was poured into four volumes of water and extracted with ether. The organic layer was washed three times with water, dried over Na_2SO_4, and concentrated under vacuum to give the product (922 mg, 95%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, 2H, J=9.0 Hz), 6.85 (d, 2H, J=9.0 Hz), 0.98 (s, 9H), 0.22 (s, 6H).

4.3. Preparation of solid-attached phenylacetylene 10

Fmoc-NH-SAL resin (1 g, loading 0.61 mmol/g) was shaken with 20% piperidine in DMF (10 ml) for 1 h. The resin was washed with DMF, MeOH, and DCM (10 ml each). Bromophenol blue test indicated the successful Fmoc-deprotection. After drying in vacuo, the resin was divided into two equal portions. Each resin portion was swollen in DMF (10 ml, 10 min), and after draining the solvent, *m*-ethynylbenzoic acid (145 mg, 0.9 mmol), 1-hydroxybenzotriazole (140 mg, 0.9 mmol), *N*,*N*′-diisopropylcarbodiimide (0.2 ml, 1.2 mmol) and DMF (15 ml) were added. After shaking for 24 h, the resins were washed with DMF, MeOH, and DCM (5×10 ml each).

4.4. Preparation of solid-attached phenylacetylene 11

Fmoc-NH-SAL resin (1 g, loading 0.61 mmol/g) was shaken with 20% piperidine in DMF (10 ml) for 1 h. The resin was washed with DMF, MeOH, and DCM (10 ml each). Bromophenol blue test indicated the successful Fmoc-deprotection. After drying in vacuo, the resin was divided into two equal portions. Each resin portion was swollen in DMF (10 ml, 10 min), and after draining the solvent, *p*-ethynylbenzoic acid (145 mg, 0.9 mmol), 1-hydroxybenzotriazole (140 mg, 0.9 mmol), *N*,*N*′-diisopropylcarbodiimide (0.2 ml, 1.2 mmol), and DMF (15 ml) were added. After shaking for 24 h, the resins were washed with DMF, MeOH, and DCM (5×10 ml each).

4.5. General procedure for the preparation of 3,5-diphenyl isoxazoles 1a–j and 2a–j

Twenty portions of the solid-attached phenylacetylenes **10** and **11** (50 mg, 0.03 mmol) were shaken with triethylamine (0.04 ml) in DCM (1 ml) for 5 min. Solutions of benzohydroximoyl chlorides **12a–j** (0.24 mmol each) in DCM (1 ml each) were added to the resin portions and the mixtures were shaken for 24 h at room temperature. The resins were washed with DMF, MeOH, and DCM (5×10 ml each). The solid-attached isoxazoles with pyridine (14 ml, 0.18 mmol) and trifluoromethanesulfonic anhydride (10 ml, 0.06 mmol) in DCM (1.5 ml) were shaken for 3 h. After filtering off the resins, the filtrates were concentrated, and purified by column chromatography on SiO_2 (100% DCM) to give the isoxazoles **1a–j** and **2a–j** as white solid.

4.5.1. 3-(4-Methoxyphenyl)-5-(3-cyanophenyl)-isoxazole 1a. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.11 (s, 1H), 8.07 (d, 1H, J=7.8 Hz), 7.81 (d, 2H, J=9.0 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.63 (t, 1H, J=7.8 Hz), 7.02 (d, 2H, J=9.0 Hz), 6.88 (s, 1H), 3.88 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.6, 162.8, 161.3, 133.2, 130.0, 129.7, 129.2, 128.7, 128.2, 121.0, 118.0, 114.4, 113.5, 98.7, 55.4; IR (KBr)

3121, 2925, 2852, 2229, 1609, 1528, 1433, 1387, 1293, 1255, 1177 cm^{-1}; HRMS (EI^+) calcd for $C_{17}H_{12}N_2O_2$: 276.0899, found: 276.0895. Anal. Calcd for $C_{17}H_{12}N_2O_2$: C, 73.90; H, 4.38; N, 10.14. Found: C, 73.85; H, 4.70; N, 9.83.

4.5.2. 3-(4-Ethoxyphenyl)-5-(3-cyanophenyl)-isoxazole 1b. ^1H NMR (300 MHz, $CDCl_3$) δ 8.10 (s, 1H), 8.06 (d, 1H, J=7.8 Hz), 7.78 (d, 2H, J=9.0 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.61 (t, 1H, J=7.8 Hz), 6.99 (d, 2H, J=9.0 Hz), 6.87 (s, 1H), 4.01 (q, 2H, J=6.9 Hz), 1.45 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 162.9, 160.7, 133.2, 130.0, 129.7, 129.2, 128.8, 128.2, 120.8, 118.0, 114.9, 113.5, 98.7, 63.6, 14.8; IR (KBr) 3123, 2981, 2925, 2226, 1613, 1529, 1440, 1387, 1293, 1252, 1177 cm^{-1}; HRMS (EI^+) calcd for $C_{18}H_{14}N_2O_2$: 290.1055, found: 290.1057. Anal. Calcd for $C_{18}H_{14}N_2O_2\cdot 0.1$hexane: C, 74.68; H, 5.15; N, 9.42. Found: C, 74.40; H, 4.95; N, 9.03.

4.5.3. 3-(4-Propoxyphenyl)-5-(3-cyanophenyl)-isoxazole 1c. ^1H NMR (300 MHz, $CDCl_3$) δ 8.10 (s, 1H), 8.07 (d, 1H, J=7.8 Hz), 7.79 (d, 2H, J=9.0 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.63 (t, 1H, J=7.8 Hz), 7.00 (d, 2H, J=9.0 Hz), 6.87 (s, 1H), 3.99 (t, 2H, J=6.6 Hz), 1.79–1.91 (m, 2H), 1.06 (t, 3H, J=7.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.6, 162.9, 160.9, 133.2, 130.0, 129.7, 129.2, 128.8, 128.2, 120.8, 118.0, 115.0, 113.6, 98.7, 69.7, 22.5, 10.5; IR (KBr) 3101, 2964, 2876, 2233, 1612, 1529, 1439, 1387, 1293, 1252, 1180 cm^{-1}; HRMS (EI^+) calcd for $C_{19}H_{16}N_2O_2$: 304.1212, found: 304.1210. Anal. Calcd for $C_{19}H_{16}N_2O_2\cdot 0.4$toluene$\cdot 0.2H_2O$: C, 75.94; H, 5.73; N, 8.12. Found: C, 75.58; H, 5.55; N, 8.04.

4.5.4. 3-(4-Butoxyphenyl)-5-(3-cyanophenyl)-isoxazole 1d. ^1H NMR (300 MHz, $CDCl_3$) δ 8.10 (s, 1H), 8.07 (d, 1H, J=7.8 Hz), 7.79 (d, 2H, J=9.0 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.63 (t, 1H, J=7.8 Hz), 7.00 (d, 2H, J=9.0 Hz), 6.87 (s, 1H), 4.02 (t, 2H, J=6.6 Hz), 1.76–1.83 (m, 2H), 1.48–1.58 (m, 2H), 1.06 (t, 3H, J=7.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 162.9, 160.9, 133.2, 130.0, 129.7, 129.2, 128.8, 128.2, 120.7, 118.0, 114.9, 113.5, 98.7, 67.8, 31.2, 19.2, 13.8; IR (KBr) 3116, 2963, 2941, 2876, 2232, 1610, 1527, 1433, 1390, 1295, 1255, 1178 cm^{-1}; HRMS (EI^+) calcd for $C_{20}H_{18}N_2O_2$: 318.1368, found: 318.1371. Anal. Calcd for $C_{20}H_{18}N_2O_2\cdot 0.05CH_2Cl_2$: C, 74.64; H, 5.65; N, 8.68. Found: C, 74.47; H, 5.31; N, 8.46.

4.5.5. 3-(4-Pentyloxyphenyl)-5-(3-cyanophenyl)-isoxazole 1e. ^1H NMR (300 MHz, $CDCl_3$) δ 8.10 (s, 1H), 8.06 (d, 1H, J=7.8 Hz), 7.78 (d, 2H, J=8.4 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.62 (t, 1H, J=7.8 Hz), 7.00 (d, 2H, J=8.4 Hz), 6.87 (s, 1H), 4.02 (t, 2H, J=6.6 Hz), 1.77–1.87 (m, 2H), 1.37–1.49 (m, 4H), 0.94 (t, 3H, J=7.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 162.9, 160.9, 133.2, 130.0, 129.7, 129.2, 128.8, 128.2, 120.7, 118.0, 114.9, 113.5, 98.7, 68.2, 28.9, 28.2, 22.4, 14.0; IR (KBr) 3103, 2972, 2936, 2869, 2232, 1609, 1530, 1431, 1390, 1296, 1261, 1178 cm^{-1}; HRMS (EI^+) calcd for $C_{21}H_{20}N_2O_2$: 332.1525, found: 332.1523. Anal. Calcd for $C_{21}H_{20}N_2O_2\cdot 0.05CH_2Cl_2$: C, 75.10; H, 6.02; N, 8.32. Found: C, 75.45; H, 6.16; N, 7.93.

4.5.6. 3-(4-Hexyloxyphenyl)-5-(3-cyanophenyl)-isoxazole 1f. ^1H NMR (300 MHz, $CDCl_3$) δ 8.10 (s, 1H), 8.06 (d, 1H, J=7.8 Hz), 7.78 (d, 2H, J=8.7 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.62 (t, 1H, J=7.8 Hz), 6.99 (d, 2H, J=8.7 Hz), 6.87 (s, 1H), 4.01 (t, 2H, J=6.6 Hz), 1.77–1.84 (m, 2H), 1.32–1.50 (m, 6H), 0.91 (t, 3H, J=7.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 162.9, 160.9, 133.2, 130.0, 129.7, 129.2, 128.8, 128.2, 120.7, 118.0, 114.9, 113.5, 98.7, 68.2, 31.6, 29.1, 25.7, 22.6, 14.0; IR (KBr) 3126, 2952, 2924, 2855, 2230, 1611, 1529, 1438, 1389, 1293, 1254, 1178 cm^{-1}; HRMS (EI^+) calcd for $C_{22}H_{22}N_2O_2$: 346.1681, found: 346.1680. Anal. Calcd for $C_{22}H_{22}N_2O_2\cdot 0.2$hexane: C, 76.62; H, 6.87; N, 7.70. Found: C, 76.43; H, 7.14; N, 7.75.

4.5.7. 3-(4-Heptyloxyphenyl)-5-(3-cyanophenyl)-isoxazole 1g. ^1H NMR (300 MHz, $CDCl_3$) δ 8.11 (s, 1H), 8.07 (d, 1H, J=7.8 Hz), 7.79 (d, 2H, J=8.7 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.63 (t, 1H, J=7.8 Hz), 7.00 (d, 2H, J=8.7 Hz), 6.87 (s, 1H), 4.02 (t, 2H, J=6.6 Hz), 1.77–1.86 (m, 2H), 1.29–1.50 (m, 8H), 0.90 (t, 3H, J=7.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 162.9, 160.9, 133.2, 130.0, 129.7, 129.2, 128.8, 128.2, 120.7, 118.0, 115.0, 113.5, 98.7, 68.2, 31.9, 29.2, 29.0, 26.0, 22.6, 14.1; IR (KBr) 3124, 2961, 2912, 2854, 2234, 1612, 1530, 1436, 1392, 1301, 1264, 1177 cm^{-1}; HRMS (EI^+) calcd for $C_{23}H_{24}N_2O_2$: 360.1838, found: 360.1843. Anal. Calcd for $C_{23}H_{24}N_2O_2\cdot 0.1$hexane: C, 76.70; H, 6.85; N, 7.68. Found: C, 76.54; H, 7.15; N, 7.46.

4.5.8. 3-(4-Octyloxyphenyl)-5-(3-cyanophenyl)-isoxazole 1h. ^1H NMR (300 MHz, $CDCl_3$) δ 8.10 (s, 1H), 8.06 (d, 1H, J=7.8 Hz), 7.78 (d, 2H, J=8.7 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.63 (t, 1H, J=7.8 Hz), 6.99 (d, 2H, J=8.7 Hz), 6.87 (s, 1H), 4.01 (t, 2H, J=6.6 Hz), 1.79–1.84 (m, 2H), 1.29–1.50 (m, 10H), 0.89 (t, 3H, J=7.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 162.9, 160.9, 133.2, 130.0, 129.7, 129.2, 128.8, 128.2, 120.7, 118.0, 115.0, 113.6, 98.7, 68.2, 31.8, 29.3, 29.2×2, 26.0, 22.7, 14.1; IR (KBr) 3123, 2960, 2921, 2854, 2236, 1616, 1533, 1437, 1390, 1300, 1268, 1178 cm^{-1}; HRMS (EI^+) calcd for $C_{24}H_{26}N_2O_2$: 374.1994, found: 374.1998. Anal. Calcd for $C_{24}H_{26}N_2O_2$: C, 76.98; H, 7.00; N, 7.48. Found: C, 76.75; H, 7.27; N, 7.20.

4.5.9. 3-(4-Nonyloxyphenyl)-5-(3-cyanophenyl)-isoxazole 1i. ^1H NMR (300 MHz, $CDCl_3$) δ 8.11 (s, 1H), 8.06 (d, 1H, J=7.8 Hz), 7.78 (d, 2H, J=8.7 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.62 (t, 1H, J=7.8 Hz), 6.99 (d, 2H, J=8.7 Hz), 6.87 (s, 1H), 4.01 (t, 2H, J=6.6 Hz), 1.78–1.83 (m, 2H), 1.28–1.50 (m, 12H), 0.90 (t, 3H, J=7.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 162.9, 160.9, 133.2, 130.0, 129.7, 129.2, 128.8, 128.2, 120.7, 118.0, 115.0, 113.5, 98.7, 68.2, 31.9, 29.5, 29.4, 29.2×2, 26.0, 22.7, 14.1; IR (KBr) 3121, 2958, 2922, 2850, 2235, 1613, 1532, 1436, 1390, 1296, 1263, 1176 cm^{-1}; HRMS (EI^+) calcd for $C_{25}H_{28}N_2O_2$: 388.2151, found: 388.2150. Anal. Calcd for $C_{25}H_{28}N_2O_2\cdot 0.2$hexane: C, 77.56; H, 7.65; N, 6.90. Found: C, 77.38; H, 7.76; N, 6.62.

4.5.10. 3-(4-Decyloxyphenyl)-5-(3-cyanophenyl)-isoxazole 1j. ^1H NMR (300 MHz, $CDCl_3$) δ 8.11 (s, 1H), 8.06 (d, 1H, J=7.8 Hz), 7.78 (d, 2H, J=8.7 Hz), 7.73 (d, 1H, J=7.8 Hz), 7.62 (t, 1H, J=7.8 Hz), 6.99 (d, 2H, J=8.7 Hz), 6.87 (s, 1H), 4.01 (t, 2H, J=6.6 Hz), 1.77–1.86 (m, 2H), 1.28–1.54 (m, 14H), 0.88 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 162.9, 160.9, 133.2, 130.0, 129.7, 129.2, 128.8, 128.2, 120.7, 118.0, 115.0, 113.5,

98.7, 68.2, 31.9, 29.5, 29.4, 29.3, 29.2, 26.0, 22.7, 14.1; IR (KBr) 3131, 2953, 2916, 2850, 2232, 1611, 1531, 1436, 1390, 1299, 1262, 1177 cm^{-1}; HRMS (EI^+) calcd for $C_{26}H_{30}N_2O_2$: 402.2307, found: 402.2306. Anal. Calcd for $C_{26}H_{30}N_2O_2$: C, 77.58; H, 7.51; N, 6.96. Found: C, 77.39; H, 7.69; N, 6.83.

4.5.11. 3-(4-Methoxyphenyl)-5-(4-cyanophenyl)-isoxazole 2a. ^1H NMR (300 MHz, $CDCl_3$) δ 7.94 (d, 2H, J=8.7 Hz), 7.80 (d, 2H, J=9.0 Hz), 7.79 (d, 2H, J=8.7 Hz), 7.01 (d, 2H, J=9.0 Hz), 6.91 (s, 1H), 3.88 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.9, 162.9, 161.3, 132.8, 131.2, 128.2, 126.2, 121.0, 118.2, 114.4, 113.5, 99.4, 55.4; IR (KBr) 3109, 2963, 2840, 2230, 1611, 1530, 1500, 1431, 1297, 1254, 1178 cm^{-1}; HRMS (EI^+) calcd for $C_{17}H_{12}N_2O_2$: 276.0899, found: 276.0899. Anal. Calcd for $C_{17}H_{12}N_2O_2 \cdot 0.25H_2O$: C, 72.72; H, 4.49; N, 9.98. Found: C, 72.98; H, 4.42; N, 9.80.

4.5.12. 3-(4-Ethoxyphenyl)-5-(4-cyanophenyl)-isoxazole 2b. ^1H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, 2H, J=8.7 Hz), 7.76–7.81 (m, 4H), 6.99 (d, 2H, J=8.7 Hz), 6.90 (s, 1H), 4.10 (q, 2H, J=6.9 Hz), 1.45 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 160.7, 132.8, 131.3, 128.2, 126.2, 120.8, 118.2, 114.9, 113.5, 99.4, 63.6, 14.7; IR (KBr) 3107, 2997, 2938, 2886, 2231, 1611, 1529, 1499, 1437, 1296, 1254, 1179 cm^{-1}; HRMS (EI^+) calcd for $C_{18}H_{14}N_2O_2$: 290.1055, found: 290.1059. Anal. Calcd for $C_{18}H_{14}N_2O_2$: C, 74.47; H, 4.86; N, 9.65. Found: C, 74.63; H, 4.66; N, 9.62.

4.5.13. 3-(4-Propoxyphenyl)-5-(4-cyanophenyl)-isoxazole 2c. ^1H NMR (300 MHz, $CDCl_3$) δ 7.94 (d, 2H, J=9.0 Hz), 7.77–7.81 (m, 4H), 7.00 (d, 2H, J=9.0 Hz), 6.91 (s, 1H), 3.99 (t, 2H, J=6.6 Hz), 1.79–1.91 (m, 2H), 1.06 (t, 3H, J=7.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 160.9, 132.8, 131.3, 128.2, 126.2, 120.7, 118.2, 115.0, 113.5, 99.4, 69.6, 22.5, 10.5; IR (KBr) 3109, 2973, 2933, 2876, 2229, 1611, 1530, 1498, 1432, 1298, 1261, 1176 cm^{-1}; HRMS (EI^+) calcd for $C_{19}H_{16}N_2O_2$: 304.1212, found: 304.1206. Anal. Calcd for $C_{19}H_{16}N_2O_2$: C, 74.98; H, 5.30; N, 9.20. Found: C, 74.78; H, 4.92; N, 9.06.

4.5.14. 3-(4-Butoxyphenyl)-5-(4-cyanophenyl)-isoxazole 2d. ^1H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, 2H, J=8.7 Hz), 7.77–7.81 (m, 4H), 6.99 (d, 2H, J=8.7 Hz), 6.91 (s, 1H), 4.02 (t, 2H, J=6.6 Hz), 1.75–1.85 (m, 2H), 1.48–1.58 (m, 2H), 0.99 (t, 3H, J=7.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 160.9, 132.8, 131.3, 128.2, 126.2, 120.7, 118.2, 115.0, 113.5, 99.4, 67.9, 31.2, 19.2, 13.8; IR (KBr) 3110, 2959, 2926, 2874, 2228, 1611, 1531, 1499, 1433, 1297, 1260, 1177 cm^{-1}; HRMS (EI^+) calcd for $C_{20}H_{18}N_2O_2$: 318.1368, found: 318.1364. Anal. Calcd for $C_{20}H_{18}N_2O_2$: C, 75.45; H, 5.70; N, 8.80. Found: C, 75.81; H, 5.59; N, 8.75.

4.5.15. 3-(4-Pentyloxyphenyl)-5-(4-cyanophenyl)-isoxazole 2e. ^1H NMR (300 MHz, $CDCl_3$) δ 7.94 (d, 2H, J=8.7 Hz), 7.77–7.80 (m, 4H), 6.99 (d, 2H, J=8.7 Hz), 6.91 (s, 1H), 4.02 (t, 2H, J=6.6 Hz), 1.80–1.85 (m, 2H), 1.38–1.48 (m, 4H), 0.94 (t, 3H, J=7.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 160.9, 132.8, 131.3, 128.2, 126.2, 120.7, 118.2, 115.0, 113.5, 99.4, 68.2, 28.9, 28.2, 22.4, 14.0; IR (KBr) 3139, 2962, 2926, 2858, 2224, 1607, 1531, 1499, 1434, 1290, 1252, 1181 cm^{-1}; HRMS (EI^+) calcd for $C_{21}H_{20}N_2O_2$: 332.1525, found: 332.1524. Anal. Calcd for $C_{21}H_{20}N_2O_2 \cdot 0.2MeCN$: C, 75.46; H, 6.10; N, 9.05. Found: C, 75.32; H, 5.75; N, 9.17.

4.5.16. 3-(4-Hexyloxyphenyl)-5-(4-cyanophenyl)-isoxazole 2f. ^1H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, 2H, J=8.7 Hz), 7.77–7.80 (m, 4H), 6.99 (d, 2H, J=8.7 Hz), 6.91 (s, 1H), 4.01 (t, 2H, J=6.6 Hz), 1.76–1.86 (m, 2H), 1.32–1.50 (m, 6H), 0.91 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 160.9, 132.8, 131.3, 128.2, 126.2, 120.7, 118.2, 115.0, 113.5, 99.4, 68.2, 31.6, 29.1, 25.7, 22.6, 14.0; IR (KBr) 3135, 2953, 2920, 2855, 2228, 1611, 1528, 1501, 1431, 1288, 1254, 1173 cm^{-1}; HRMS (EI^+) calcd for $C_{22}H_{22}N_2O_2$: 346.1681, found: 346.1681. Anal. Calcd for $C_{22}H_{22}N_2O_2$: C, 76.28; H, 6.40; N, 8.09. Found: C, 76.21; H, 6.72; N, 7.98.

4.5.17. 3-(4-Heptyloxyphenyl)-5-(4-cyanophenyl)-isoxazole 2g. ^1H NMR (300 MHz, $CDCl_3$) δ 7.94 (d, 2H, J=8.7 Hz), 7.76–7.80 (m, 4H), 6.99 (d, 2H, J=8.7 Hz), 6.91 (s, 1H), 4.01 (t, 2H, J=6.6 Hz), 1.77–1.86 (m, 2H), 1.31–1.57 (m, 8H), 0.90 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 160.9, 132.8, 131.3, 128.2, 126.2, 120.7, 118.2, 115.0, 113.5, 99.4, 68.2, 31.8, 29.2, 29.0, 26.0, 22.6, 14.1; IR (KBr) 3136, 2943, 2925, 2869, 2228, 1612, 1531, 1498, 1433, 1294, 1256, 1179 cm^{-1}; HRMS (EI^+) calcd for $C_{23}H_{24}N_2O_2$: 360.1838, found: 360.1835. Anal. Calcd for $C_{23}H_{24}N_2O_2 \cdot 0.25$hexane: C, 77.03; H, 7.26; N, 7.33. Found: C, 76.93; H, 7.13; N, 7.69.

4.5.18. 3-(4-Octyloxyphenyl)-5-(4-cyanophenyl)-isoxazole 2h. ^1H NMR (300 MHz, $CDCl_3$) δ 7.94 (d, 2H, J=8.7 Hz), 7.76–7.81 (m, 4H), 6.99 (d, 2H, J=8.7 Hz), 6.91 (s, 1H), 4.01 (t, 2H, J=6.6 Hz), 1.76–1.86 (m, 2H), 1.29–1.47 (m, 10H), 0.89 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 160.9, 132.8, 131.3, 128.2, 126.2, 120.7, 118.2, 115.0, 113.5, 99.4, 68.2, 31.8, 29.3, 29.2×2, 26.0, 22.7, 14.1; IR (KBr) 3099, 2953, 2925, 2855, 2228, 1610, 1530, 1498, 1430, 1293, 1255, 1177 cm^{-1}; HRMS (EI^+) calcd for $C_{24}H_{26}N_2O_2$: 374.1994, found: 374.1989. Anal. Calcd for $C_{24}H_{26}N_2O_2 \cdot 0.125CH_2Cl_2$: C, 75.24; H, 6.87; N, 7.27. Found: C, 74.91; H, 7.24; N, 6.96.

4.5.19. 3-(4-Nonyloxyphenyl)-5-(4-cyanophenyl)-isoxazole 2i. ^1H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, 2H, J=8.7 Hz), 7.76–7.81 (m, 4H), 6.99 (d, 2H, J=8.7 Hz), 6.90 (s, 1H), 4.01 (t, 2H, J=6.6 Hz), 1.76–1.86 (m, 2H), 1.28–1.49 (m, 12H), 0.88 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 160.9, 132.8, 131.3, 128.2, 126.2, 120.7, 118.2, 115.0, 113.5, 99.4, 68.2, 31.9, 29.5, 29.4, 29.2×2, 26.0, 22.7, 14.1; IR (KBr) 3118, 2952, 2917, 2853, 2240, 1617, 1531, 1504, 1441, 1295, 1250, 1178 cm^{-1}; HRMS (EI^+) calcd for $C_{25}H_{28}N_2O_2$: 388.2151, found: 388.2151. Anal. Calcd for $C_{25}H_{28}N_2O_2$: C, 77.29; H, 7.26; N, 7.21. Found: C, 77.26; H, 7.52; N, 7.06.

4.5.20. 3-(4-Decyloxyphenyl)-5-(4-cyanophenyl)-isoxazole 2j. ^1H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, 2H, J=8.7 Hz), 7.76–7.81 (m, 4H), 6.99 (d, 2H, J=8.7 Hz), 6.91 (s, 1H), 4.01 (t, 2H, J=6.6 Hz), 1.76–1.83 (m, 2H), 1.28–

1.49 (m, 14H), 0.88 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 160.9, 132.8, 131.3, 128.2, 126.2, 120.7, 118.2, 115.0, 113.5, 99.4, 68.2, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 26.0, 22.7, 14.1; IR (KBr) 3121, 2955, 2920, 2851, 2234, 1611, 1528, 1502, 1437, 1292, 1254, 1177 cm^{-1}; HRMS (EI$^+$) calcd for $C_{26}H_{30}N_2O_2$: 402.2307, found: 402.2305. Anal. Calcd for $C_{26}H_{30}N_2O_2$: C, 77.89; H, 7.96; N, 6.61. Found: C, 77.95; H, 7.92; N, 6.88.

4.6. General procedure for the preparation of 3,5-diphenyl isoxazoles 3a–j, 4a–j, 5a–j, and 6a–j

Forty portions of the solid-attached phenylacetylene **11** (50 mg, 0.03 mmol) were shaken with triethylamine (0.04 ml) in DCM (1 ml) for 5 min. Solutions of 4-[(*tert*-butyldimethylsilyl)oxy]benzohydroximoyl chloride **13** (0.24 mmol each) in DCM (1 ml each) were added to the resin portions and the mixtures were shaken for 24 h at room temperature. The resins were washed with DMF, MeOH, and DCM (5×10 ml each). After drying in vacuo, the resins were swollen in THF (1 ml, 30 min each). THF solutions of TBAF (24 mg, 0.09 ml each) were added to the resin portions, and the mixtures were stirred for 1 h. The resins were washed with DMF, MeCN, and DCM (5× 10 ml each). Benzoic acids **17a–j**,[19] **18a–j**,[20] **19a–j**,[21] and **20a–j**[22] (0.24 mmol each), *N*,*N*′-diisopropylcarbodiimide (0.05 ml, 0.3 mmol each), DMAP (11 mg, 0.09 mmol each), and DMF (1.5 ml each) were added to the resin portions. After shaking for 24 h at room temperature, the resins were washed with DMF, MeCN, and DCM (5×10 ml each). The polymer-attached isoxazoles with pyridine (14 μl, 0.18 mmol) and trifluoromethanesulfonic anhydride (10 μl, 0.06 mmol) in DCM (1.5 ml) were shaken for 4 h. After filtering off the resins, the filtrates were concentrated, and purified by column chromatography on SiO_2 (100% DCM) to give the isoxazoles **3a–j**, **4a–j**, **5a–j**, and **6a–j** as white solid.

4.6.1. 3-[4-(4-Methoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3a. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.18 (d, 2H, J=9.0 Hz), 7.92–7.98 (m, 4H), 7.80 (d, 2H, J= 8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 7.01 (d, 2H, J=9.0 Hz), 6.97 (s, 1H), 3.91 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.3, 164.6, 164.1, 162.6, 152.8, 132.9, 132.4, 131.0, 128.1, 126.3, 126.1, 122.6, 121.5, 118.1, 114.0, 113.8, 99.6, 55.5; IR (KBr) 3127, 2975, 2924, 2847, 2229, 1731, 1604, 1510, 1433, 1258, 1213, 1163 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{24}H_{17}N_2O_4$ (M+H$^+$): 397.1188, found: 397.1187. Anal. Calcd for $C_{24}H_{16}N_2O_2\cdot 0.2CH_2Cl_2\cdot 0.2H_2O$: C, 70.85; H, 4.49; N, 6.51. Found: C, 71.09; H, 4.17; N, 6.28.

4.6.2. 3-[4-(4-Ethoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3b. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, 2H, J=9.0 Hz), 7.91–7.97 (m, 4H), 7.80 (d, 2H, J= 9.0 Hz), 7.36 (d, 2H, J=9.0 Hz), 6.99 (d, 2H, J=9.0 Hz), 6.97 (s, 1H), 4.14 (q, 2H, J=6.9 Hz), 1.47 (t, 3H, J= 6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.7, 163.5, 162.6, 152.7, 132.9, 132.4, 131.1, 128.1, 126.3, 126.0, 122.6, 121.2, 118.2, 114.4, 113.7, 99.6, 63.9, 14.7; IR (KBr) 3124, 2983, 2936, 2224, 1734, 1606, 1509, 1432, 1257, 1215, 1166 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{25}H_{19}N_2O_4$ (M+H$^+$): 411.1345, found: 411.1343. Anal. Calcd for $C_{25}H_{18}N_2O_2$: C, 73.16; H, 4.42; N, 6.83. Found: C, 72.82; H, 4.78; N, 7.09.

4.6.3. 3-[4-(4-Propoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3c. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, 2H, J=9.0 Hz), 7.91–7.98 (m, 4H), 7.80 (d, 2H, J= 9.0 Hz), 7.36 (d, 2H, J=9.0 Hz), 6.99 (d, 2H, J=9.0 Hz), 6.97 (s, 1H), 4.02 (t, 2H, J=6.6 Hz), 1.83–1.90 (m, 2H), 1.07 (t, 3H, J=7.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.3, 164.6, 163.8, 162.6, 152.8, 132.9, 132.4, 131.1, 128.1, 126.3, 126.0, 122.6, 121.2, 118.1, 114.4, 113.7, 99.6, 69.8, 22.5, 10.4; IR (KBr) 3127, 2965, 2872, 2226, 1720, 1602, 1509, 1436, 1257, 1213, 1172 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{26}H_{21}N_2O_4$ (M+H$^+$): 425.1501, found: 425.1504. Anal. Calcd for $C_{26}H_{20}N_2O_2\cdot 0.05CH_2Cl_2$: C, 72.98; H, 4.73; N, 6.53. Found: C, 72.58; H, 4.78; N, 6.92.

4.6.4. 3-[4-(4-butoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3d. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, 2H, J=9.3 Hz), 7.96 (d, 2H, J=8.7 Hz), 7.93 (d, 2H, J= 8.7 Hz), 7.80 (d, 2H, J=9.0 Hz), 7.36 (d, 2H, J=9.0 Hz), 6.99 (d, 2H, J=9.3 Hz), 6.97 (s, 1H), 4.06 (t, 2H, J= 6.6 Hz), 1.77–1.87 (m, 2H), 1.46–1.59 (m, 2H), 1.00 (t, 3H, J=7.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.7, 163.8, 162.6, 152.7, 132.9, 132.4, 131.1, 128.1, 126.3, 126.0, 122.6, 121.1, 118.2, 114.4, 113.7, 99.6, 68.0, 31.1, 19.2, 13.8; IR (KBr) 3126, 2944, 2875, 2226, 1721, 1602, 1509, 1435, 1257, 1213, 1173 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{27}H_{23}N_2O_4$ (M+H$^+$): 439.1658, found: 439.1662. Anal. Calcd for $C_{27}H_{22}N_2O_2$: C, 73.96; H, 5.06; N, 6.39. Found: C, 73.98; H, 5.16; N, 6.28.

4.6.5. 3-[4-(4-Pentyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3e. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, 2H, J=9.0 Hz), 7.96 (d, 2H, J=8.7 Hz), 7.92 (d, 2H, J= 8.7 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.35 (d, 2H, J=8.7 Hz), 6.99 (d, 2H, J=9.0 Hz), 6.97 (s, 1H), 4.05 (t, 2H, J= 6.6 Hz), 1.79–1.88 (m, 2H), 1.37–1.50 (m, 4H), 0.95 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 164.7, 163.7, 162.6, 152.7, 132.9, 132.4, 131.1, 128.0, 126.3, 126.0, 122.6, 121.1, 118.2, 114.4, 113.7, 99.6, 68.3, 28.8, 28.1, 22.4, 14.0; IR (KBr) 3128, 2947, 2872, 2228, 1720, 1603, 1511, 1435, 1260, 1216, 1174 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{28}H_{25}N_2O_4$ (M+H$^+$): 453.1814, found: 453.1815. Anal. Calcd for $C_{28}H_{24}N_2O_2\cdot 0.1$MeCN: C, 74.18; H, 5.36; N, 6.44. Found: C, 74.28; H, 4.98; N, 6.63.

4.6.6. 3-[4-(4-Hexyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3f. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, 2H, J=9.0 Hz), 7.96 (d, 2H, J=8.7 Hz), 7.93 (d, 2H, J= 8.7 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 6.99 (d, 2H, J=9.0 Hz), 6.97 (s, 1H), 4.06 (t, 2H, J= 6.6 Hz), 1.78–1.88 (m, 2H), 1.33–1.51 (m, 6H), 0.92 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.7, 163.8, 162.6, 152.7, 132.9, 132.4, 131.1, 128.1, 126.3, 126.0, 122.6, 121.1, 118.1, 114.4, 113.7, 99.6, 68.4, 31.5, 29.0, 25.6, 22.6, 14.0; IR (KBr) 3127, 2920, 2874, 2227, 1721, 1604, 1510, 1435, 1257, 1214, 1173 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{29}H_{27}N_2O_4$ (M+H$^+$): 467.1971, found: 467.1973. Anal. Calcd for $C_{29}H_{26}N_2O_2\cdot 0.25H_2O$: C, 73.95; H, 5.65; N, 5.95. Found: C, 73.79; H, 5.85; N, 5.76.

4.6.7. 3-[4-(4-Heptyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3g. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (d, 2H, J=9.0 Hz), 7.95 (d, 2H, J=8.7 Hz), 7.92 (d, 2H, J= 8.7 Hz), 7.79 (d, 2H, J=8.7 Hz), 7.35 (d, 2H, J=8.7 Hz),

6.98 (d, 2H, J=9.0 Hz), 6.96 (s, 1H), 4.05 (t, 2H, J=6.6 Hz), 1.78–1.87 (m, 2H), 1.32–1.51 (m, 8H), 0.90 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 164.6, 163.7, 162.6, 152.7, 132.9, 132.3, 131.1, 128.0, 126.3, 126.0, 122.6, 121.1, 118.1, 114.4, 113.7, 99.6, 68.4, 31.7, 29.1, 29.0, 25.9, 22.6, 14.1; IR (KBr) 3126, 2921, 2850, 2225, 1721, 1602, 1509, 1436, 1257, 1213, 1173 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{30}H_{29}N_2O_4$ (M+H$^+$): 481.2127, found: 481.2126. Anal. Calcd for $C_{30}H_{28}N_2O_2$: C, 74.98; H, 5.87; N, 5.83. Found: C, 74.80; H, 5.99; N, 5.78.

4.6.8. 3-[4-(4-Octyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3h. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, 2H, J=9.0 Hz), 7.96 (d, 2H, J=8.7 Hz), 7.93 (d, 2H, J=8.7 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 6.97–7.00 (m, 3H), 4.05 (t, 2H, J=6.6 Hz), 1.78–1.88 (m, 2H), 1.25–1.48 (m, 10H), 0.90 (t, 3H, J=6.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.7, 163.7, 162.6, 152.7, 132.9, 132.4, 131.1, 128.1, 126.3, 126.0, 122.6, 121.1, 118.2, 114.4, 113.7, 99.6, 68.4, 31.8, 29.3, 29.2, 29.1, 26.0, 22.6, 14.1; IR (KBr) 3127, 2919, 2851, 2227, 1721, 1604, 1509, 1436, 1257, 1213, 1173 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{31}H_{31}N_2O_4$ (M+H$^+$): 495.2284, found: 495.2283. Anal. Calcd for $C_{31}H_{30}N_2O_2\cdot 0.5H_2O$: C, 73.94; H, 6.20; N, 5.56. Found: C, 73.92; H, 6.03; N, 5.82.

4.6.9. 3-[4-(4-Nonyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3i. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (d, 2H, J=8.7 Hz), 7.96 (d, 2H, J=8.7 Hz), 7.92 (d, 2H, J=8.7 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.35 (d, 2H, J=8.7 Hz), 6.97–7.00 (m, 3H), 4.05 (t, 2H, J=6.6 Hz), 1.78–1.85 (m, 2H), 1.28–1.48 (m, 12H), 0.89 (t, 3H, J=6.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 164.7, 163.7, 162.5, 152.7, 132.9, 132.3, 131.0, 128.0, 126.3, 126.0, 122.6, 121.0, 118.2, 114.3, 113.6, 99.6, 68.3, 31.9, 29.5, 29.3, 29.2, 29.1, 26.0, 22.7, 14.1; IR (KBr) 3127, 2918, 2848, 2233, 1729, 1607, 1510, 1434, 1260, 1209, 1173 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{32}H_{32}N_2O_4$ (M+H$^+$): 509.2440, found: 509.2442. Anal. Calcd for $C_{32}H_{31}N_2O_2\cdot 0.2$MeCN: C, 75.30; H, 6.36; N, 5.96. Found: C, 75.41; H, 6.10; N, 5.97.

4.6.10. 3-[4-(4-Decyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 3j. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, 2H, J=9.0 Hz), 7.96 (d, 2H, J=8.7 Hz), 7.93 (d, 2H, J=8.7 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 6.99 (d, 2H, J=9.0 Hz), 6.97 (s, 1H), 4.05 (t, 2H, J=6.6 Hz), 1.78–1.85 (m, 2H), 1.29–1.49 (m, 14H), 0.89 (t, 3H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 164.6, 163.7, 162.6, 152.7, 132.9, 132.3, 131.1, 128.0, 126.3, 126.0, 122.6, 121.1, 118.1, 114.4, 113.7, 99.6, 68.4, 31.9, 29.5, 29.3×2, 29.1, 26.0, 22.7, 14.1; IR (KBr) 3127, 2918, 2850, 2227, 1721, 1603, 1509, 1436, 1258, 1213, 1173 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{33}H_{35}N_2O_4$ (M+H$^+$): 523.2597, found: 523.2607. Anal. Calcd for $C_{32}H_{31}N_2O_2$: C, 75.84; H, 6.56; N, 5.36. Found: C, 75.77; H, 6.62; N, 5.24.

4.6.11. 3-[4-(3,4-Dimethoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4a. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.96 (d, 2H, J=8.7 Hz), 7.93 (d, 2H, J=8.7 Hz), 7.88 (dd, 1H, J=8.4, 2.1 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.68 (d, 1H, J=2.1 Hz), 7.36 (d, 2H, J=8.7 Hz), 6.97 (d, 1H, J=8.4 Hz), 6.97 (s, 1H), 3.99 (s, 3H), 3.98 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.7, 162.5, 153.8, 152.7, 148.9, 132.9, 131.1, 128.1, 126.3, 126.1, 124.6, 122.6, 121.5, 118.1, 113.7, 112.4, 110.4, 99.6, 56.1×2; IR (KBr) 3120, 2922, 2850, 2229, 1712, 1595, 1519, 1436, 1278, 1212, 1167, 1143 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{25}H_{19}N_2O_5$ (M+H$^+$): 427.1294, found: 427.1298. Anal. Calcd for $C_{25}H_{18}N_2O_2$: C, 70.42; H, 4.25; N, 6.57. Found: C, 70.21; H, 4.43; N, 6.49.

4.6.12. 3-[4-(3,4-Diethoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4b. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91–7.97 (m, 4H), 7.84 (dd, 1H, J=8.4, 2.1 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.68 (d, 1H, J=2.1 Hz), 7.35 (d, 2H, J=8.7 Hz), 6.94–6.97 (m, 2H), 4.15–4.24 (m, 4H), 1.48–1.54 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.7, 162.6, 153.6, 152.7, 148.3, 132.9, 131.1, 128.1, 126.3, 126.1, 124.5, 122.6, 121.2, 118.1, 114.2, 113.7, 111.7, 99.6, 64.7, 64.6, 14.7, 14.6; IR (KBr) 3126, 2983, 2939, 2230, 1720, 1599, 1521, 1434, 1281, 1213, 1170, 1133 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{27}H_{23}N_2O_5$ (M+H$^+$): 455.1607, found: 455.1606. Anal. Calcd for $C_{27}H_{22}N_2O_2\cdot 0.25H_2O$: C, 70.65; H, 4.94; N, 6.10. Found: C, 70.60; H, 5.04; N, 6.10.

4.6.13. 3-[4-(3,4-Dipropoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4c. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91–7.97 (m, 4H), 7.83 (dd, 1H, J=8.4, 2.1 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.68 (d, 1H, J=2.1 Hz), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 6.95 (d, 1H, J=8.4 Hz), 4.03–4.09 (m, 4H), 1.83–1.97 (m, 4H), 1.05–1.11 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 154.0, 152.7, 148.7, 132.9, 131.1, 128.0, 126.3, 126.0, 124.5, 122.6, 121.1, 118.1, 114.7, 113.7, 112.0, 99.6, 70.8, 70.5, 22.5, 22.4, 10.5, 10.4; IR (KBr) 3128, 2967, 2878, 2225, 1726, 1596, 1519, 1432, 1279, 1199, 1170, 1142 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{29}H_{27}N_2O_5$ (M+H$^+$): 483.1920, found: 483.1915. Anal. Calcd for $C_{29}H_{26}N_2O_2$: C, 72.18; H, 5.43; N, 5.81. Found: C, 72.23; H, 5.54; N, 5.77.

4.6.14. 3-[4-(3,4-Dibutoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4d. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.83 (dd, 1H, J=8.4, 1.8 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.67 (d, 1H, J=1.8 Hz), 7.36 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 6.95 (d, 1H, J=8.4 Hz), 4.07–4.13 (m, 4H), 1.80–1.91 (m, 4H), 1.47–1.59 (m, 4H), 0.97–1.03 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 154.1, 152.7, 148.7, 132.9, 131.1, 128.1, 126.3, 126.0, 124.5, 122.6, 121.1, 118.1, 114.6, 113.7, 111.9, 99.6, 69.1, 68.8, 31.2, 31.1, 19.2, 19.1, 13.9, 13.8; IR (KBr) 3128, 2957, 2873, 2225, 1726, 1596, 1519, 1432, 1281, 1200, 1170, 1144 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{31}H_{31}N_2O_5$ (M+H$^+$): 511.2233, found: 511.2233. Anal. Calcd for $C_{31}H_{30}N_2O_2$: C, 72.92; H, 5.92; N, 5.49. Found: C, 73.07; H, 5.98; N, 5.44.

4.6.15. 3-[4-(3,4-Dipentyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4e. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.83 (dd, 1H, J=8.4, 2.1 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.67 (d, 1H, J=2.1 Hz), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 6.94 (d, 1H, J=8.4 Hz), 4.06–4.12 (m, 4H), 1.84–1.91 (m, 4H), 1.37–1.52 (m, 8H), 0.92–0.97 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 154.1, 152.8, 148.7, 132.9, 131.1, 128.1, 126.3,

126.1, 124.5, 122.6, 121.1, 118.2, 114.6, 113.7, 111.9, 99.6, 69.4, 69.1, 28.8, 28.7, 28.2, 28.1, 22.4, 14.0; IR (KBr) 3128, 2955, 2858, 2227, 1720, 1595, 1522, 1430, 1284, 1207, 1171, 1146 cm^{-1}; HRMS (FAB^+) calcd for $C_{33}H_{35}N_2O_5$ ($M+H^+$): 539.2546, found: 539.2546. Anal. Calcd for $C_{33}H_{34}N_2O_2$: C, 73.58; H, 6.36; N, 5.20. Found: C, 73.61; H, 6.55; N, 5.09.

4.6.16. 3-[4-(3,4-Dihexyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4f. ^1H NMR (300 MHz, $CDCl_3$) δ 7.91–7.98 (m, 4H), 7.83 (dd, 1H, J=8.7, 2.1 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.67 (d, 1H, J=2.1 Hz), 7.36 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 6.95 (d, 1H, J=8.7 Hz), 4.06–4.12 (m, 4H), 1.81–1.92 (m, 4H), 1.34–1.53 (m, 12H), 0.89–0.94 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 154.1, 152.8, 148.7, 132.9, 131.1, 128.1, 126.3, 126.0, 124.5, 122.6, 121.1, 118.1, 114.6, 113.7, 111.9, 99.6, 69.4, 69.1, 31.5, 29.1, 29.0, 25.7, 22.6, 14.0; IR (KBr) 3128, 2931, 2857, 2227, 1720, 1595, 1523, 1431, 1284, 1209, 1170, 1147 cm^{-1}; HRMS (FAB^+) calcd for $C_{35}H_{39}N_2O_5$ ($M+H^+$): 567.2859, found: 567.2856. Anal. Calcd for $C_{35}H_{38}N_2O_2 \cdot 0.2MeCN$: C, 73.96; H, 6.77; N, 5.36. Found: C, 73.87; H, 6.68; N, 5.40.

4.6.17. 3-[4-(3,4-Diheptyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4g. ^1H NMR (300 MHz, $CDCl_3$) δ 7.91–7.98 (m, 4H), 7.83 (dd, 1H, J=8.7, 2.1 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.67 (d, 1H, J=2.1 Hz), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 6.94 (d, 1H, J=8.7 Hz), 4.05–4.12 (m, 4H), 1.81–1.92 (m, 4H), 1.25–1.54 (m, 16H), 0.87–0.92 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 154.0, 152.7, 148.7, 132.9, 131.1, 128.1, 126.3, 126.0, 124.5, 122.6, 121.1, 118.1, 114.6, 113.7, 111.9, 99.6, 69.4, 69.1, 31.8, 29.2, 29.0, 26.0, 25.9, 22.6, 14.1; IR (KBr) 3128, 2931, 2855, 2227, 1720, 1595, 1523, 1431, 1284, 1207, 1170, 1147 cm^{-1}; HRMS (FAB^+) calcd for $C_{37}H_{43}N_2O_5$ ($M+H^+$): 595.3172, found: 595.3193. Anal. Calcd for $C_{37}H_{42}N_2O_2$: C, 74.72; H, 7.12; N, 4.71. Found: C, 74.67; H, 7.13; N, 4.66.

4.6.18. 3-[4-(3,4-Dioctyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4h. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.83 (dd, 1H, J=8.7, 2.1 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.67 (d, 1H, J=2.1 Hz), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 6.95 (d, 1H, J=8.7 Hz), 4.05–4.12 (m, 4H), 1.81–1.92 (m, 4H), 1.29–1.54 (m, 20H), 0.86–0.90 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 154.0, 152.7, 148.7, 132.9, 131.1, 128.1, 126.3, 126.0, 124.5, 122.6, 121.1, 118.12, 114.6, 113.7, 111.9, 99.6, 69.4, 69.1, 31.8, 29.3, 29.2, 29.1, 29.0×3, 22.7, 14.1; IR (KBr) 3128, 2919, 2847, 2226, 1720, 1595, 1523, 1431, 1284, 1206, 1170, 1148 cm^{-1}; HRMS (FAB^+) calcd for $C_{39}H_{47}N_2O_5$ ($M+H^+$): 623.3485, found: 623.3481. Anal. Calcd for $C_{39}H_{46}N_2O_2$: C, 75.21; H, 7.44; N, 4.50. Found: C, 75.13; H, 7.38; N, 4.40.

4.6.19. 3-[4-(3,4-Dinonyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4i. ^1H NMR (300 MHz, $CDCl_3$) δ 7.91–7.98 (m, 4H), 7.83 (dd, 1H, J=8.7, 2.1 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.67 (d, 1H, J=2.1 Hz), 7.35 (d, 2H, J=8.7 Hz), 6.98 (s, 1H), 6.94 (d, 1H, J=8.7 Hz), 4.05–4.11 (m, 4H), 1.81–1.92 (m, 4H), 1.28–1.58 (m, 24H), 0.85–0.90 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 164.8, 162.6, 154.0, 152.7, 148.6, 132.9, 131.0, 128.0, 126.3, 126.0, 124.5, 122.6, 121.0, 118.2, 114.4, 113.6, 111.8, 99.6, 69.3, 69.0, 31.9, 29.6, 29.4, 29.3, 29.1, 29.0, 26.0, 25.9, 22.7, 14.1; IR (KBr) 3128, 2918, 2847, 2227, 1720, 1596, 1523, 1431, 1285, 1205, 1170, 1148 cm^{-1}; HRMS (FAB^+) calcd for $C_{41}H_{51}N_2O_5$ ($M+H^+$): 651.3798, found: 651.3792. Anal. Calcd for $C_{41}H_{50}N_2O_2$: C, 75.66; H, 7.74; N, 4.30. Found: C, 75.70; H, 7.58; N, 4.20.

4.6.20. 3-[4-(3,4-Didecyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 4j. ^1H NMR (300 MHz, $CDCl_3$) δ 7.91–7.97 (m, 4H), 7.83 (dd, 1H, J=8.7, 2.1 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.67 (d, 1H, J=2.1 Hz), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 6.94 (d, 1H, J=8.7 Hz), 4.05–4.12 (m, 4H), 1.81–1.90 (m, 4H), 1.28–1.49 (m, 24H), 0.85–0.91 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 154.1, 152.8, 148.7, 132.9, 131.1, 128.1, 126.3, 126.1, 124.5, 122.6, 121.1, 118.1, 114.7, 113.7, 112.0, 99.6, 69.4, 69.1, 31.9, 29.6×2, 29.4×2, 29.3, 29.2, 29.0, 26.0×2, 22.7, 14.1; IR (KBr) 3128, 2918, 2848, 2227, 1721, 1595, 1523, 1431, 1285, 1208, 1170, 1148 cm^{-1}; HRMS (FAB^+) calcd for $C_{43}H_{55}N_2O_5$ ($M+H^+$): 679.4111, found: 679.4128. Anal. Calcd for $C_{43}H_{54}N_2O_2$: C, 76.07; H, 8.02; N, 4.13. Found: C, 76.29; H, 8.38; N, 4.36.

4.6.21. 3-[4-(3,5-Dimethoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5a. ^1H NMR (300 MHz, $CDCl_3$) δ 7.96 (d, 2H, J=8.7 Hz), 7.94 (d, 2H, J=8.7 Hz), 7.80 (d, 2H, J=8.4 Hz), 7.35–7.38 (m, 4H), 6.97 (s, 1H), 6.74 (t, 1H, J=2.1 Hz), 3.88 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.3, 164.7, 162.5, 160.9, 152.6, 132.9, 131.1, 131.0, 128.1, 126.3×2, 122.5, 118.1, 113.7, 107.8, 106.6, 99.6, 55.7; IR (KBr) 3126, 2981, 2954, 2230, 1738, 1613, 1504, 1428, 1355, 1308, 1209, 1160 cm^{-1}; HRMS (FAB^+) calcd for $C_{25}H_{19}N_2O_5$ ($M+H^+$): 427.1294, found: 427.1276. Anal. Calcd for $C_{25}H_{18}N_2O_2$: C, 70.42; H, 4.25; N, 6.57. Found: C, 70.63; H, 4.34; N, 6.44.

4.6.22. 3-[4-(3,5-Diethoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5b. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92–7.97 (m, 4H), 7.80 (d, 2H, J=8.7 Hz), 7.35 (d, 2H, J=8.7 Hz), 7.33 (d, 2H, J=2.4 Hz), 6.97 (s, 1H), 6.73 (t, 1H, J=2.4 Hz), 4.09 (q, 4H, J=6.9 Hz), 1.44 (t, 6H, J=6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 160.1, 152.5, 132.9, 131.1, 130.1, 128.1, 126.3, 126.2, 122.5, 118.1, 113.7, 108.3, 107.3, 99.6, 63.9, 14.7; IR (KBr) 3126, 2983, 2926, 2881, 2225, 1730, 1593, 1504, 1439, 1348, 1300, 1204, 1169 cm^{-1}; HRMS (FAB^+) calcd for $C_{27}H_{23}N_2O_5$ ($M+H^+$): 455.1607, found: 455.1604. Anal. Calcd for $C_{27}H_{22}N_2O_2$: C, 71.35; H, 4.88; N, 6.16. Found: C, 71.56; H, 5.06; N, 6.11.

4.6.23. 3-[4-(3,5-Dipropoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5c. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.81 (d, 2H, J=8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 7.33 (d, 2H, J=2.1 Hz), 6.97 (s, 1H), 6.74 (t, 1H, J=2.1 Hz), 3.98 (t, 4H, J=6.6 Hz), 1.78–1.90 (m, 4H), 1.06 (t, 6H, J=7.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 160.4, 152.6, 132.9, 131.1, 130.8, 128.1, 126.3×2, 122.5, 118.1, 113.8, 108.3, 107.4, 99.6, 70.0, 22.5, 10.5; IR (KBr) 3126, 2965, 2877, 2225, 1730, 1593, 1504, 1436, 1353, 1296, 1199, 1173 cm^{-1}; HRMS (FAB^+) calcd for $C_{29}H_{27}N_2O_5$ ($M+H^+$): 483.1920,

found: 483.1921. Anal. Calcd for $C_{29}H_{26}N_2O_2$: C, 72.18; H, 5.43; N, 5.81. Found: C, 72.22; H, 5.57; N, 5.83.

4.6.24. 3-[4-(3,5-Dibutoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5d. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.80 (d, 2H, J=8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 7.33 (d, 2H, J=2.4 Hz), 6.97 (s, 1H), 6.73 (t, 1H, J=2.4 Hz), 4.02 (t, 4H, J=6.6 Hz), 1.75–1.84 (m, 4H), 1.45–1.56 (m, 4H), 0.99 (t, 6H, J=7.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 160.4, 152.6, 132.9, 131.1, 130.8, 128.1, 126.3, 126.2, 122.5, 118.1, 113.7, 108.3, 107.3, 99.6, 68.1, 31.2, 19.2, 13.8; IR (KBr) 3129, 2957, 2872, 2224, 1733, 1592, 1500, 1438, 1350, 1301, 1209, 1171 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{31}H_{31}N_2O_5$ (M+H$^+$): 511.2233, found: 511.2231. Anal. Calcd for $C_{31}H_{30}N_2O_2$: C, 72.92; H, 5.92; N, 5.49. Found: C, 72.93; H, 6.02; N, 5.47.

4.6.25. 3-[4-(3,5-dipentyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5e. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.80 (d, 2H, J=8.7 Hz), 7.35 (d, 2H, J=8.7 Hz), 7.32 (d, 2H, J=2.4 Hz), 6.97 (s, 1H), 6.73 (t, 1H, J=2.4 Hz), 4.01 (t, 4H, J=6.6 Hz), 1.76–1.86 (m, 4H), 1.36–1.49 (m, 8H), 0.94 (t, 6H, J=6.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 160.3, 152.5, 132.9, 131.0, 130.7, 128.1, 126.3, 126.2, 122.5, 118.1, 113.7, 108.2, 107.2, 99.6, 68.4, 28.8, 28.1, 22.4, 14.0; IR (KBr) 3127, 2957, 2858, 2232, 1733, 1592, 1501, 1450, 1350, 1303, 1200, 1163 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{33}H_{35}N_2O_5$ (M+H$^+$): 539.2546, found: 539.2540. Anal. Calcd for $C_{33}H_{34}N_2O_2$: C, 73.58; H, 6.36; N, 5.20. Found: C, 73.61; H, 6.55; N, 5.09.

4.6.26. 3-[4-(3,5-Dihexyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5f. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.81 (d, 2H, J=8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 7.32 (d, 2H, J=2.4 Hz), 6.97 (s, 1H), 6.73 (t, 1H, J=2.4 Hz), 4.01 (t, 4H, J=6.6 Hz), 1.76–1.85 (m, 4H), 1.32–1.50 (m, 12H), 0.91 (t, 6H, J=6.9 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.4, 164.8, 162.6, 160.4, 152.6, 132.9, 131.1, 130.8, 128.1, 126.3×2, 122.5, 118.1, 113.8, 108.3, 107.3, 99.6, 68.5, 31.6, 29.1, 25.7, 22.6, 14.0; IR (KBr) 3119, 2953, 2856, 2227, 1735, 1596, 1503, 1450, 1350, 1301, 1200, 1163 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{35}H_{39}N_2O_5$ (M+H$^+$): 567.2859, found: 567.2866. Anal. Calcd for $C_{35}H_{38}N_2O_2$: C, 74.18; H, 6.76; N, 4.94. Found: C, 74.19; H, 6.97; N, 4.94.

4.6.27. 3-[4-(3,5-Diheptyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5g. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.80 (d, 2H, J=8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 7.32 (d, 2H, J=2.4 Hz), 6.97 (s, 1H), 6.73 (t, 1H, J=2.4 Hz), 4.01 (t, 4H, J=6.6 Hz), 1.76–1.85 (m, 4H), 1.32–1.47 (m, 16H), 0.90 (t, 6H, J=6.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 160.3, 152.6, 132.9, 131.1, 130.8, 128.1, 126.3, 126.3, 122.5, 118.2, 113.7, 108.2, 107.3, 99.6, 68.4, 31.8, 29.2, 29.0, 26.0, 22.6, 14.1; IR (KBr) 3120, 2923, 2854, 2227, 1735, 1596, 1503, 1450, 1351, 1301, 1200, 1172 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{37}H_{43}N_2O_5$ (M+H$^+$): 595.3172, found: 595.3163. Anal. Calcd for $C_{37}H_{42}N_2O_2 \cdot 0.25$MeCN: C, 74.45; H, 7.12; N, 5.21. Found: C, 74.50; H, 7.34; N, 5.22.

4.6.28. 3-[4-(3,5-Dioctyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5h. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.81 (d, 2H, J=8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 7.32 (d, 2H, J=2.4 Hz), 6.97 (s, 1H), 6.73 (t, 1H, J=2.4 Hz), 4.00 (t, 4H, J=6.6 Hz), 1.76–1.83 (m, 4H), 1.25–1.47 (m, 20H), 0.89 (t, 6H, J=6.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.4, 164.9, 162.4, 160.4, 152.7, 132.9, 131.1, 130.8, 128.1, 126.3×2, 122.5, 118.2, 113.8, 108.3, 107.5, 99.6, 68.5, 31.8, 29.3, 29.2×2, 26.0, 22.7, 14.1; IR (KBr) 3121, 2921, 2851, 2230, 1734, 1595, 1503, 1450, 1351, 1302, 1200, 1160 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{39}H_{47}N_2O_5$ (M+H$^+$): 623.3485, found: 623.3491. Anal. Calcd for $C_{39}H_{46}N_2O_2$: C, 75.21; H, 7.44; N, 4.50. Found: C, 75.11; H, 7.57; N, 4.50.

4.6.29. 3-[4-(3,5-Dinonyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5i. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.81 (d, 2H, J=8.7 Hz), 7.36 (d, 2H, J=8.7 Hz), 7.32 (d, 2H, J=2.4 Hz), 6.97 (s, 1H), 6.73 (t, 1H, J=2.4 Hz), 4.01 (t, 4H, J=6.6 Hz), 1.76–1.85 (m, 4H), 1.28–1.47 (m, 24H), 0.88 (t, 6H, J=6.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 160.4, 152.6, 132.9, 131.1, 130.8, 128.1, 126.3, 126.3, 122.5, 118.1, 113.7, 108.3, 107.3, 99.6, 68.5, 31.9, 29.5, 29.4, 29.2×2, 26.0, 22.7, 14.1; IR (KBr) 3124, 2922, 2852, 2230, 1734, 1595, 1505, 1450, 1350, 1302, 1200, 1160 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{41}H_{51}N_2O_5$ (M+H$^+$): 651.3798, found: 651.3795. Anal. Calcd for $C_{41}H_{50}N_2O_2$: C, 75.66; H, 7.74; N, 4.30. Found: C, 75.55; H, 7.72; N, 4.30.

4.6.30. 3-[4-(3,5-Didecyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 5j. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.97 (m, 4H), 7.80 (d, 2H, J=8.7 Hz), 7.35 (d, 2H, J=8.7 Hz), 7.32 (d, 2H, J=2.4 Hz), 6.97 (s, 1H), 6.73 (t, 1H, J=2.4 Hz), 4.01 (t, 4H, J=6.6 Hz), 1.75–1.85 (m, 4H), 1.28–1.47 (m, 28H), 0.88 (t, 6H, J=6.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 160.3, 152.5, 132.9, 131.0, 130.7, 128.1, 126.3×2, 122.5, 118.2, 113.7, 108.2, 107.2, 99.6, 68.4, 31.9, 29.6, 29.5, 29.3×2, 29.1, 26.0, 22.7, 14.1; IR (KBr) 3123, 2921, 2851, 2229, 1733, 1594, 1503, 1450, 1350, 1302, 1201, 1159 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{43}H_{55}N_2O_5$ (M+H$^+$): 679.4111, found: 679.4116. Anal. Calcd for $C_{43}H_{54}N_2O_2$: C, 76.07; H, 8.02; N, 4.13. Found: C, 75.82; H, 8.05; N, 4.39.

4.6.31. 3-[4-(3,4,5-Trimethoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6a. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.96 (d, 2H, J=8.7 Hz), 7.95 (d, 2H, J=8.7 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.47 (s, 2H), 7.36 (d, 2H, J=8.7 Hz), 6.98 (s, 1H), 3.96 (s, 3H), 3.96 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.4, 164.6, 162.5, 153.2, 152.6, 143.1, 132.9, 131.1, 128.1, 126.3, 126.1, 124.0, 122.6, 118.1, 113.8, 107.6, 99.6, 61.0, 56.4; IR (KBr) 3122, 2921, 2851, 2229, 1733, 1594, 1503, 1450, 1350, 1201, 1159 cm^{-1}; HRMS (FAB$^+$) calcd for $C_{26}H_{21}N_2O_6$ (M+H$^+$): 457.1400, found: 457.1418. Anal. Calcd for $C_{26}H_{20}N_2O_2$: C, 68.42; H, 4.42; N, 6.14. Found: C, 68.39; H, 4.50; N, 5.97.

4.6.32. 3-[4-(3,4,5-Triethoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6b. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.81 (d, 2H, J=8.7 Hz), 7.43 (s, 2H), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 4.12–4.22 (m, 6H), 1.47 (t, 6H, J=7.2 Hz), 1.39 (t, 3H, J=7.2 Hz); ^{13}C NMR

(75 MHz, $CDCl_3$) δ 168.3, 164.7, 162.5, 143.0, 152.8, 152.6, 132.9, 131.1, 128.1, 126.3, 126.2, 123.6, 122.6, 118.1, 113.7, 108.7, 99.6, 69.1, 64.9, 15.6, 14.8; IR (KBr) 3109, 2980, 2929, 2229, 1735, 1593, 1500, 1435, 1340, 1194, 1133 cm^{-1}; HRMS (FAB^+) calcd for $C_{29}H_{27}N_2O_6$ ($M+H^+$): 499.1869, found: 499.1864. Anal. Calcd for $C_{29}H_{26}N_2O_2$: C, 69.87; H, 5.26; N, 5.62. Found: C, 69.72; H, 5.34; N, 5.56.

4.6.33. 3-[4-(3,4,5-Tripropoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6c. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.80 (d, 2H, J=8.7 Hz), 7.43 (s, 2H), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 4.01–4.08 (m, 6H), 1.74–1.93 (m, 6H), 1.04–1.10 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 153.0, 152.6, 143.2, 132.9, 131.1, 128.1, 126.3, 126.2, 123.5, 122.6, 118.1, 113.7, 108.7, 99.6, 75.2, 70.8, 23.5, 22.6, 10.6, 10.5; IR (KBr) 3125, 2965, 2876, 2231, 1732, 1590, 1502, 1433, 1336, 1196, 1122 cm^{-1}; HRMS (FAB^+) calcd for $C_{32}H_{33}N_2O_6$ ($M+H^+$): 541.2339, found: 541.2339. Anal. Calcd for $C_{32}H_{32}N_2O_2$: C, 71.09; H, 5.97; N, 5.18. Found: C, 71.09; H, 6.08; N, 5.14.

4.6.34. 3-[4-(3,4,5-Tributoxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6d. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.81 (d, 2H, J=8.7 Hz), 7.42 (s, 2H), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 4.05–4.10 (m, 6H), 1.71–1.88 (m, 6H), 1.47–1.59 (m, 6H), 0.88–1.02 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 153.0, 152.7, 143.2, 132.9, 131.1, 128.1, 126.3, 126.2, 123.5, 122.6, 118.1, 113.7, 108.6, 99.6, 73.2, 69.0, 32.3, 31.3, 19.3, 19.1, 13.9, 13.8; IR (KBr) 3129, 2931, 2871, 2231, 1731, 1592, 1504, 1432, 1337, 1195, 1128 cm^{-1}; HRMS (FAB^+) calcd for $C_{35}H_{39}N_2O_6$ ($M+H^+$): 583.2808, found: 583.2811. Anal. Calcd for $C_{35}H_{38}N_2O_2$: C, 72.14; H, 6.57; N, 4.81. Found: C, 72.35; H, 6.61; N, 4.81.

4.6.35. 3-[4-(3,4,5-Tripentyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6e. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.80 (d, 2H, J=8.7 Hz), 7.42 (s, 2H), 7.35 (d, 2H, J=8.7 Hz), 6.98 (s, 1H), 4.03–4.10 (m, 6H), 1.73–1.89 (m, 6H), 1.37–1.51 (m, 12H), 0.91–0.96 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 153.0, 152.6, 143.2, 132.9, 131.0, 128.1, 126.3, 126.2, 123.5, 122.6, 118.1, 113.7, 108.6, 99.6, 73.6, 69.2, 30.0, 28.9, 28.2, 28.1, 22.5, 22.4, 14.1, 14.0; IR (KBr) 3131, 2936, 2871, 2237, 1733, 1588, 1500, 1432, 1341, 1200, 1116 cm^{-1}; HRMS (FAB^+) calcd for $C_{38}H_{45}N_2O_6$ ($M+H^+$): 625.3278, found: 625.3276. Anal. Calcd for $C_{38}H_{44}N_2O_2$: C, 73.05; H, 7.10; N, 4.48. Found: C, 73.02; H, 7.25; N, 4.33.

4.6.36. 3-[4-(3,4,5-Trihexyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6f. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.81 (d, 2H, J=8.7 Hz), 7.42 (s, 2H), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 4.04–4.09 (m, 6H), 1.72–1.89 (m, 6H), 1.25–1.54 (m, 18H), 0.89–0.93 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 153.0, 152.7, 143.2, 132.9, 131.1, 128.1, 126.3, 126.2, 123.5, 122.6, 118.1, 113.7, 108.6, 99.6, 73.6, 69.3, 31.7, 31.5, 30.3, 29.2, 25.7×2, 22.7, 22.6, 14.1, 14.0; IR (KBr) 3140, 2931, 2859, 2229, 1736, 1588, 1503, 1432, 1337, 1191, 1117 cm^{-1}; HRMS (FAB^+) calcd for $C_{41}H_{51}N_2O_6$ ($M+H^+$): 667.3747, found: 667.3740. Anal. Calcd for $C_{41}H_{50}N_2O_2$: C, 73.85; H, 7.56; N, 4.20. Found: C, 73.89; H, 7.73; N, 4.14.

4.6.37. 3-[4-(3,4,5-Triheptyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6g. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92–7.97 (m, 4H), 7.80 (d, 2H, J=8.7 Hz), 7.42 (s, 2H), 7.34 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 4.03–4.09 (m, 6H), 1.73–1.89 (m, 6H), 1.26–1.54 (m, 24H), 0.87–0.91 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 153.0, 152.7, 143.2, 132.9, 131.0, 128.1, 126.3, 126.2, 123.5, 122.6, 118.1, 113.7, 108.6, 99.6, 73.6, 69.3, 31.9, 31.8, 30.3, 29.3, 29.2, 29.0, 26.0×2, 22.6×2, 14.1×2; IR (KBr) 3128, 2923, 2853, 2233, 1733, 1587, 1502, 1432, 1338, 1192, 1119 cm^{-1}; HRMS (FAB^+) calcd for $C_{44}H_{57}N_2O_6$ ($M+H^+$): 709.4217, found: 709.4216. Anal. Calcd for $C_{44}H_{56}N_2O_2$: C, 74.55; H, 7.96; N, 3.95. Found: C, 74.61; H, 8.17; N, 4.12.

4.6.38. 3-[4-(3,4,5-Trioctyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6h. ^1H NMR (300 MHz, $CDCl_3$) δ 7.92–7.98 (m, 4H), 7.81 (d, 2H, J=8.7 Hz), 7.42 (s, 2H), 7.35 (d, 2H, J=8.7 Hz), 6.97 (s, 1H), 4.03–4.09 (m, 6H), 1.73–1.89 (m, 6H), 1.25–1.57 (m, 30H), 0.86–0.90 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.6, 153.0, 152.7, 143.2, 132.9, 131.0, 128.1, 126.3, 126.2, 123.5, 122.6, 118.6, 113.7, 108.6, 99.6, 73.6, 69.3, 31.9, 31.8, 30.3, 29.5, 29.3×2, 26.1, 22.7×2, 14.1; IR (KBr) 3137, 2954, 2854, 2227, 1732, 1588, 1500, 1431, 1339, 1202, 1119 cm^{-1}; HRMS (FAB^+) calcd for $C_{47}H_{63}N_2O_6$ ($M+H^+$): 751.4686, found: 751.4686. Anal. Calcd for $C_{47}H_{62}N_2O_2 \cdot 0.5MeCN \cdot 0.5EtOAc$: C, 73.63; H, 8.34; N, 4.29. Found: C, 74.68; H, 8.55; N, 4.14.

4.6.39. 3-[4-(3,4,5-Trinonyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6i. ^1H NMR (300 MHz, $CDCl_3$) δ 7.96 (d, 2H, J=8.4 Hz), 7.93 (d, 2H, J=8.4 Hz), 7.80 (d, 2H, J=8.7 Hz), 7.42 (s, 2H), 7.34 (d, 2H, J=8.7 Hz), 6.98 (s, 1H), 4.03–4.09 (m, 6H), 1.72–1.88 (m, 6H), 1.28–1.49 (m, 36H), 0.85–0.90 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 153.0, 152.6, 143.1, 132.9, 131.0, 128.1, 126.3, 126.1, 123.4, 122.6, 118.2, 113.7, 108.5, 99.6, 73.6, 69.2, 31.9×2, 30.3, 29.7, 29.6, 29.4, 29.3, 26.1, 26.0, 22.7×2, 14.1; IR (KBr) 3136, 2925, 2854, 2226, 1732, 1588, 1500, 1432, 1339, 1202, 1119 cm^{-1}; HRMS (FAB^+) calcd for $C_{50}H_{69}N_2O_6$ ($M+H^+$): 793.5156, found: 793.5160. Anal. Calcd for $C_{47}H_{62}N_2O_2 \cdot 0.5MeCN$: C, 75.29; H, 8.61; N, 4.30. Found: C, 75.14; H, 8.81; N, 4.40.

4.6.40. 3-[4-(3,4,5-Tridecyloxybenzoyloxy)phenyl]-5-(4-cyanophenyl)-isoxazole 6j. ^1H NMR (300 MHz, $CDCl_3$) δ 7.96 (d, 2H, J=8.4 Hz), 7.94 (d, 2H, J=8.4 Hz), 7.81 (d, 2H, J=8.7 Hz), 7.42 (s, 2H), 7.35 (d, 2H, J=8.7 Hz), 6.98 (s, 1H), 4.03–4.09 (m, 6H), 1.72–1.88 (m, 6H), 1.27–1.49 (m, 42H), 0.85–0.90 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.3, 164.8, 162.5, 153.0, 152.6, 143.2, 132.9, 131.0, 128.1, 126.3, 126.2, 123.4, 122.6, 118.1, 113.7, 108.5, 99.6, 73.6, 69.3, 31.9×2, 30.3, 29.7×2, 29.6×2, 29.4, 29.3×2, 26.1, 26.0, 22.7, 14.1; IR (KBr) 3130, 2920, 2851, 2234, 1736, 1588, 1502, 1433, 1338, 1191, 1120 cm^{-1}; HRMS (FAB^+) calcd for $C_{53}H_{75}N_2O_6$ ($M+H^+$): 835.5625, found: 835.5632. Anal. Calcd for $C_{47}H_{62}N_2O_2$: C, 76.22; H, 8.93; N, 3.35. Found: C, 76.55; H, 8.88; N, 3.62.

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