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Synthesis of POSS-Functionalized Imidazole

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ABSTRACT. The synthesis of *N*-substituted POSS-imidazole in one pot typically prepared using the condensation method from diketone and aminopropylisobutyl-POSS. A wide variety of functional groups and substitution patterns were tolerated under the present procedure. The resulting compounds can be used as valuable products allowing for the elaboration to OLED, DSSC building block.

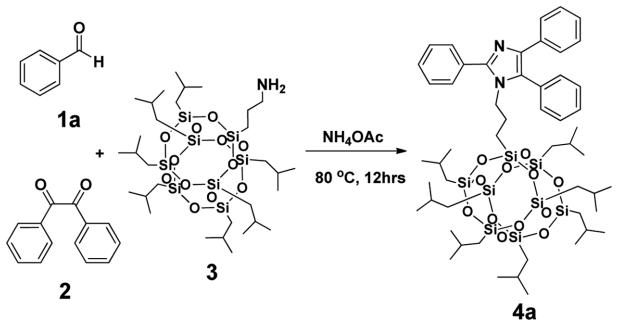
Key words: Polyhedral oligomeric silsesquioxane (POSS), Imidazoles, OLEDs, DSSCs

INTRODUCTION

The imidazole ring represent an important class of heterocycle frequently found in many natural product.[1] In addition, imidazole derivative have been reported to possess pharmacological properties[2] such as antiplasmodium,[3] antitumor,[4] and antifungal,[5] and acting as glucagon receptors,[6] therapeutic agents,[7] antibacterial agents,[8] and inhibitors of p38 MAP kinase[9] and B-Raf kinase,[10] plant growth regulators[11] and fungicides.[12] On the other hand, highly substituted imidazole derivative possess a good photophysical property, which result in their potential in material chemistry application such as organic electroluminescent device (OLED).[13] The use of the 1*H*-phenanthro[9,10-*d*]-imidazol-2-yl group because of its supramolecular property has been reported in a molecular tweezer system for explosives[14] and as a fluorophore in a superradiant laser dye[15] as a result of its photophysical property. Triarylamine-containing imidazole[16] or fused aromatic imidazole such as phenanthroimidazole[17] and pyrenoimidazole[18] material in a solar cells have also been found to serve as promising donors when connected with oligothiophene unit. Consequently, many effort in the past decade have been focused on the preparation of those privileged scaffold. For example, the synthesis of 1,2,4,5-tetraubstuted imidazole have been developed by using various catalytic systems including I_2,[19a] BF_3–SiO_2,[19b] SBPPSA,[19c] alumina,[19d] $FeCl_3 \cdot 6H_2O$,[19e] $Cu(OAc)_2$,[19f] and DABCO.[19g] Transition-metal-catalyzed direct C–H or N–H functionalization provides a powerful tool for the formation of multisubstituted imidazole.[20]

Polyhedral oligomeric silsesquioxane (POSS) is a unique modifier to high-performance materials due to its unique three-dimensional structure.[21–22] In POSS molecule, the rigid inorganic core provide high stiffness, thermo- and photo-stability, the organic corner group offer excellent solubility, processability and compatibility with other materials. Many POSS-based functional material, such as liquid crystal (LC) material,[23–26] light-emitting material,[27–29] dental restorative material[30] and ionic liquid[31] have been prepared. And they are usually based on mono- or octa-substituted POSSs. The incorporation of POSS moieties into organic molecules or polymers may have dramatic effect on their properties, these hybrid materials display superior property to the organic material alone. Especially, aminopropylisobutyl-POSS prepared by employing a facile, corner-capping methodology is one of the most interesting functional POSS because it can be used as the precursor for many functional POSS derivative,[32] and it also can be used as organic light-emitting material,[33] fluorescent nano-sensors.[34]

As part of our further investigations of imidazole derivative and aminopropylisobutyl-POSS, we herein describe the synthesis of *N*-substituted POSS-imidazole in one pot typically prepared using the Debus–Radziszewski method from diketone such as benzil, phenanthrene-9,10-dione, pyrene-4,5-dione[35] and aminopropylisobutyl-POSS (*Scheme* 1). To the best of our knowledge, POSS-containing molecular hybrid material incorporated to nitrogen of imidazole as organic component remain unknown. Due to the excellent electric properties of these naturally occurring imidazole derivatives, incorporation of imidazole into molecular hybrids of POSS is expected to yield corresponding material with good properties, functionality, and application.



***Scheme* 1.** *N*-Substituted POSS-imidazole prepared with diketone and aminopropylisobutyl-POSS.

EXPERIMENTAL

General chemicals were purchased from commercial vendors and used as such without any purification. Reaction progress was monitored using thin layer chromatographic technique (TLC) on pre-coated aluminum sheets silica gel-60/UV254 with I_2 and UV light as detecting agents. NMR analysis was done with Bruker Avance-400 spectrometer using TMS as an internal standard.

General method for 4a-4k: Preparation of 2,4,5-triphenyl-imidazole-isobuyl-POSS (4a)

The product was prepared by refluxing benzil (1.0 g, 4.7 mmol, 1.0 eq.), benzaldehyde (0.5 g, 4.7 mmol, 1.0 eq.), propylamine isobutyl-POSS (6.2 g, 7.1 mmol, 1.5 eq.), and ammonium acetate (1.8 g, 23.6 mmol, 5.0 eq.) in glacial acetic acid (25 mL) for 12 hrs under an nitrogen atmosphere. After cooling to room temperature, a pale yellow mixture was obtained and poured into a methanol solution under stirring. The separated solid was filtered off, washed with methanol, and dried to give a pale yellow solid. The solid was purified by column chromatography (hexane/EtOAc=5:1) on silica gel. A white powder was stirred in refluxing ethanol, subsequently filtered, and dried in vacuum to give **4a-4k** (yield, 9-25%).

2,4,5-Triphenyl-imidazole-isobuyl-POSS (4a)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.22 (t, J = 8.4 Hz, 2H), 0.52 (dd, J = 6.8, 7.2 Hz, 14H), 0.90–0.95 (dd, J = 6.8, 6.4 Hz, 42H), 1.42–1.50 (m, 2H), 1.73–1.89 (m, 7H), 3.88 (t, J = 7.6 Hz, 2H), 7.11–7.70 (m, 15H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.4, 22.5, 23.8, 25.6, 25.7, 47.3, 126.2, 126.8, 128.0, 128.6, 128.8, 129.0, 129.1, 129.5, 130.9, 131.5, 134.6, 137.8, 147.6.

4,5-Diphenyl-2-p-tolyl-imidazole-isobutyl-POSS (4b)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.22 (t, J = 8.4 Hz, 2H), 0.52 (dd, J = 6.8, 7.2 Hz, 14H), 0.90–0.95 (dd, J = 6.8, 6.4 Hz, 42H), 1.42–1.50 (m, 2H), 1.73–1.89 (m, 7H), 3.88 (t, J = 7.6 Hz, 2H), 7.11–7.70 (m, 14H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.4, 22.5, 23.8, 25.6, 25.7, 47.3, 126.2, 126.8, 128.0, 128.6, 128.8, 129.0, 129.1, 129.5, 130.9, 131.5, 134.6, 137.8, 147.6.

2-(4-Fluorophenyl)-4,5-diphenyl-imidazole-isobutyl-POSS (4c)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.22 (t, J = 8.4 Hz, 2H), 0.52 (dd, J = 7.2, 7.2 Hz, 14H), 0.90–0.95 (dd, J = 6.8, 6.4 Hz, 42H), 1.41–1.49 (m, 2H), 1.72–1.89 (m, 7H), 3.85 (t, J = 7.6 Hz, 2H), 7.11–7.69 (m, 14H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.4, 22.5, 23.8, 25.6, 25.7, 47.2, 115.7, 115.9, 126.3, 126.8, 128.1, 128.7, 129.1, 129.7, 130.9, 131.4, 134.5, 137.8, 147.6.

2-(4-Chlorophenyl)-4,5-diphenyl-imidazole-isobutyl-POSS (4d)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.22 (t, J = 8.4 Hz, 2H), 0.53 (dd, J = 7.2, 7.2 Hz, 14H), 0.92 (dd, J = 6.4, 6.8 Hz, 42H), 1.41–1.49 (m, 2H), 1.72–1.87 (m, 7H), 3.86 (t, J = 7.6 Hz, 2H), 7.11–7.66 (m, 14H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.4, 22.5, 23.8, 25.6, 25.7, 47.3, 126.3, 126.8, 128.1, 128.7, 128.8, 128.9, 129.1, 129.9, 130.3, 130.9, 131.3, 134.4, 134.8, 138.0, 146.4.

2-(4-Bromophenyl)-4,5-diphenyl-imidazole-isobutyl-POSS (4e)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.22 (t, J = 8.4 Hz, 2H), 0.53 (dd, J = 7.2, 6.8 Hz, 14H), 0.92 (dd, J = 6.4, 6.8 Hz, 42H), 1.41–1.49 (m, 2H), 1.72–1.88 (m, 7H), 3.87 (t, J = 7.6 Hz, 2H), 7.12–7.63 (m, 14H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.5, 22.4, 22.5, 23.8, 25.6, 25.7, 47.4, 123.1, 126.4, 126.7, 128.1, 128.8, 129.1, 130.0, 130.4, 130.5, 130.9, 131.3, 131.9, 134.4, 138.1, 146.4.

4-(4,5-Diphenyl-imidazol-2-yl)phenol-isobutyl-POSS (4f)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.18 (t, J = 8.4 Hz, 2H), 0.52 (dd, J = 7.2, 7.2 Hz, 14H), 0.90 (dd, J = 6.8, 6.8 Hz, 42H), 1.37–1.45 (m, 2H), 1.71–1.87 (m, 7H), 3.84 (t, J = 7.6 Hz, 2H), 6.71–7.46 (m, 14H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.4, 22.5, 23.8, 25.6, 25.6, 25.7, 47.3, 116.8, 126.7, 127.4, 128.2, 128.8, 129.0, 129.2, 130.5, 130.7, 130.9, 130.9, 148.4, 158.8, 176.2.

2-(4-(Trifluoromethyl)phenyl)-4,5-diphenyl-imidazole-isobutyl-POSS (4g)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.23 (t, J = 8.8 Hz, 2H),

0.52 (dd, J = 7.2, 7.2 Hz, 14H), 0.91 (dd, J = 6.8, 6.4 Hz, 42H), 1.46–1.52 (m, 2H), 1.72–1.88 (m, 7H), 3.91 (t, J = 7.6 Hz, 2H), 7.13–7.86 (m, 14H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.4, 22.5, 23.8, 25.6, 25.7, 47.5, 125.6, 125.7, 126.5, 126.8, 128.1, 128.9, 129.2, 130.3, 130.9, 131.1, 134.3, 135.1, 138.4, 145.9.

2-(4-Methoxyphenyl)-4,5-diphenyl-imidazole-isobutyl-POSS (4h)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.22 (t, J = 8.4 Hz, 2H), 0.53 (dd, J = 6.8, 7.2 Hz, 14H), 0.93 (dd, J = 6.4, 6.4 Hz, 42H), 1.41–1.49 (m, 2H), 1.72–1.88 (m, 7H), 3.85 (t, J = 7.6 Hz, 2H), 3.87 (s, 3H), 6.98–7.62 (m, 14H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.3, 22.4, 23.8, 25.6, 25.6, 55.3, 114.1, 126.8, 128.0, 129.1, 130.5, 131.0.

2-(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-4,5-diphenyl-1*H*-imidazole-isobutyl-POSS (4i)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.20 (t, J = 8.4 Hz, 2H), 0.53 (dd, J = 6.8, 7.2 Hz, 14H), 0.91 (dd, J = 6.4, 6.8 Hz, 42H), 1.38 (s, 12H), 1.40–1.48 (m, 2H), 1.72–1.88 (m, 7H), 3.88 (t, J = 7.6 Hz, 2H), 7.13–7.92 (m, 14H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.4, 22.5, 23.8, 24.9, 25.7, 25.8, 83.9, 126.9, 128.1, 128.2, 129.1, 131.1, 135.0.

***N*-Phenyl-*N*-(4-(4,5-diphenyl-imidazol-2-yl)phenyl) benzenamine-isobutyl-POSS (4j)**

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.26 (t, J = 8.8 Hz, 2H), 0.53 (dd, J = 6.8, 7.2 Hz, 14H), 0.90 (dd, J = 6.8, 6.8 Hz, 42H), 1.46–1.54 (m, 2H), 1.72–1.88 (m, 7H), 3.88 (t, J = 7.6 Hz, 2H), 7.04–7.54 (m, 24H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.4, 22.5, 23.8, 25.6, 25.7, 122.9, 123.4, 124.9, 126.8, 128.0, 129.1, 129.9, 130.0, 147.3.

4,5-Diphenyl-2-(4-(5-(thiophen-2-yl)thiophen-2-yl)phenyl)-1*H*-imidazole-isobutyl-POSS (4k)

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.43 (t, J = 8.4 Hz, 2H), 0.56 (dd, J = 7.2, 7.2 Hz, 14H), 0.92 (dd, J = 6.4, 6.4 Hz, 42H), 1.68–1.75 (m, 2H), 1.76–1.89 (m, 7H), 3.96 (t, J = 9.0 Hz, 2H), 7.04–7.504 (m, 15H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 9.4, 22.4, 22.5, 23.9, 25.6, 25.7, 47.3, 124.1, 124.7, 125.7, 126.4, 126.8, 127.9, 128.1, 128.9, 129.1, 130.3, 131.0, 131.1, 132.2, 134.3, 137.0, 138.2, 140.9.

2-(4-Bromophenyl)-1*H*-phenanthro[9,10-*d*]imidazole isobutyl-POSS (6)

The mixture of 9,10-phenanthrenedione (0.56 g, 2.7 mmol, 1.0 eq.), bromobenzaldehyde (0.5 g, 2.7 mmol, 1.0 eq.), propylamineisobutyl-POSS (3.5 g, 4.1 mmol, 1.5 eq.) and ammonium acetate (1.04 g, 13.5 mmol, 3.1 eq.) was refluxed in glacial acetic acid (25 mL) for 12 hrs under an N_2 atmosphere. After cooling to room temperature, a pale yellow mixture was obtained and poured into a methanol solution under stirring. The separated solid was filtered off, washed with methanol, and dried to give a pale yellow solid. The solid was purified by column chromatography (hexane/EtOAc=5:1) on silica gel. A white powder was stirred in refluxing ethanol, subsequently filtered, and dried in vacuum to give **6** (0.1 g, 3%).

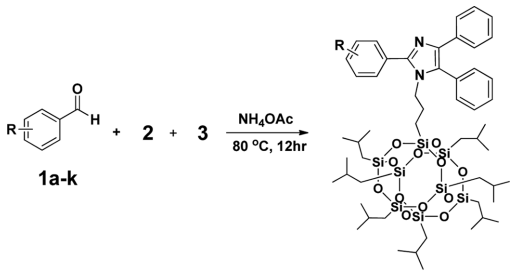
^{1}H NMR (400 MHz, $CDCl_3$): δ 0.50 (dd, J = 7.2, 7.2 Hz, 14H), 0.88 (dd, J = 6.8, 6.8 Hz, 42H), 1.67–1.885 (m, 7H), 1.99–2.07 (m, 2H), 4.56 (t, J = 7.6 Hz, 2H), 7.59–8.85 (m, 12H).

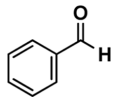
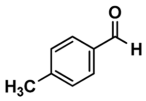
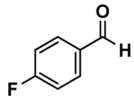
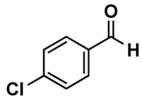
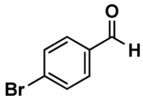
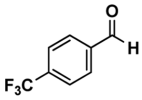
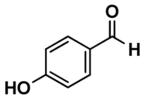
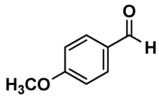
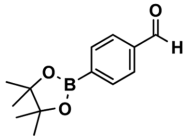
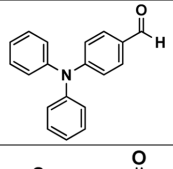
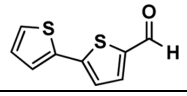
RESULTS AND DISCUSSION

With the previously conditions, we have studied the scope and limitations with various aldehyde and diketone. In *Table* 1, the one-pot approach to synthesize a wide range of imidazole derivatives with various substituents on the aromatic moieties using benzil as diketone was performed.

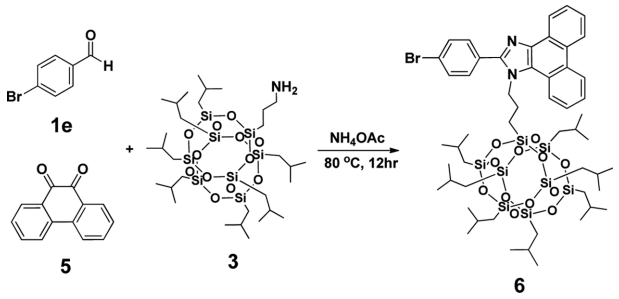
As shown in *Table* 1, the first application of the above approach to the preparation of 2,4,5-triphenyl-imidazole-isobutyl-POSS was successfully synthesized **4a-4k** in 9-25% yield. Compared with the yield of general organic synthesis, slightly lower yields, but in first synthesis of *N*-substituted POSS-imidazole in one-pot synthesis, it is the moderate yield. Treatment of benzaldehyde **1a** and amino propylisobutyl-POSS **3**, benzil **2** with ammonium acetate at reflux for 12 hrs in the presence of acetic acid by one-pot method resulted in conversion to POSS-Imidazole **4a** in 11% yield. We next investigated the scope of this three-component reaction of aldehydes bearing both electron-rich and electron-deficient aryl substituents under the same condition. In the reaction proceeded with the aldehyde bearing methyl group as electron donating group at the *p*-position of phenyl, the yield could be decreased as low as 8%, **4b**. Influence of electron-drawing halogen substituents on para-positon of the phenyl ring were also investigated in the reaction. The more electro-deficient fluorobenzaldehyde proceeded smoothly to give desired product **4c** in the moderate 25% yield. Chloro and bromobenzaldehyde afforded also desired product leading to the relatively low isolated yield of **4d-4e**. It was found that CF_3-substituted benzaldehyde can also yield the target product **4f**. A benzaldehyde **1g-1h** with an electron donating group at the para-position of the aryl ring was furnished the desired product **4g-4h** in low yields. The product substituted as 4-formyl-

***Table* 1.** Substrate scope of one-pot reaction of various benzaldehydes with benzil using aminopropylisobutyl-POSS



Entry	Aldehyde		Yield (%)[a]	
1		**1a**	**4a**	11
2		**1b**	**4b**	8
3		**1c**	**4c**	25
4		**1d**	**4d**	23
5		**1e**	**4e**	21
6		**1f**	**4f**	13
7		**1g**	**4g**	9
8		**1h**	**4h**	13
9		**1i**	**4i**	16
10		**1j**	**4j**	15
11		**1k**	**4k**	9

Reaction conditions: aldehydes (**1a-11a**, 1.0 eq.), benzil (**2**, 1.1 eq.), POSS (**3**, 2.0 eq.).
[a]Isolated yields



***Scheme* 2.** Synthesis of 2-(4-bromophenyl)-1*H*-phenan thro[9,10-*d*]imidazole isobutyl-POSS (**6**).

phenylboronic acid ester was isolated as **4i** in 16% yield. Furthermore, triphenylaminebenzaldehyde **1j** has also been utilized for this reaction. We found that desired product **4j** was formed in 15% yield under the same conditions. Dithiophenealdehyde **1k** bearing sulfur atom was also resulted in a low isolated 9% yield to give **4k.**

The 1*H*-phenanthro[9,10-d]imidazole unit shows excellent electron injection and transport properties and good thermal stability, making it a suitable component for organic semiconductors used in organic lightemitting diodes (OLEDs)[1] and solar cells.[2] Herein, in order to further study the reactivity of other diketones, we performed a multicomponent reaction under the same condition by replacing 1*H*-phenanthro[9,10-d]imidazole **5** instead of benzyl **2**. Phenanthrene-9,10-dione proceeded to give the desired product **6**, affording in a very low isolated yield (<5%), but it is a very important starting point in the developing of advanced materials (*Scheme* 2).

CONCLUSIONS

In conclusion, we developed one-pot method for the synthesis of POSS-imidazoles from aldehydes and aminopropyl-isobutyl-POSS as the corrected compound in the moderate yield (9-25%). The condensation reaction were conducted from aldehydes and amine derivative *via* the *in situ* formation of imine intermediate using NH_4OAc. The product could be applied to the synthesis of OLED, a DSSC senstizier.

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