Synthesis Characterization of Polyamide Metallodendrimers and their Catalytic Activities in Ethylene Oligomerization

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Abstract Nickel complexes with aromatic amine terminated first generation (G1) and second generation (G2) polyamide dendrimers {POA-(NH$_2$)$_n$ (n = 6 or 12, POM = Polyamide)} were synthesized. All the synthesized dendrimers and metallodendrimers were characterized by elemental and spectral analysis. These novel dendritic complexes were evaluated as catalyst precursors in the oligomerization of ethylene, using methylaluminoxane (MAO) as an activator at two different pressure and different Al/M (M = Zn) ratio. In the case of 1 atm pressure of ethylene, and 500:1 and 1,000:1 Al/Ni molar ratio, for **C$_1$** and **C$_2$** respectively display higher catalytic activity towards ethylene polymerization. Oligomerization using the generation 1 dendrimer complex, **C$_1$** yields short chain oligomers whereas those obtained from the generation 2 dendrimer catalyst, **C$_2$** resulted in longer chain products with molecular weights up to 9.54×10^4 and 9.86×10^4 g/mol at 1 and 5 atm respectively.

1 Introduction

Metallodendrimers are well-defined, highly branched, there dimensional macromolecules with characteristic globular structures for the larger systems [1]. These novel macro-molecules functionalized with transition metals have inspired many researchers to develop new materials and several applications have been explored, catalysis being one of them [2]. The recent impressive strides in synthetic procedures increased the accessibility of functionalized metallodendrimers, resulting in a rapid development of dendrimer chemistry. The potential interest of metallodendrimers as catalyst, arise mainly from their ability to combine the advantage of homogenous and heterogeneous catalyses in one system, more ever their shape and sized make them more suitable for recycling than soluble polymer supported catalyst [3–5]. The multiple sites located at their surfaces may afford reaction rates comparable to those shown in homogenous systems due to the accessibility for the substrate [6]. Recently there have been several reports detailing the application of metallodendrimers as catalysts. In a number of instances, some sort of dendritic effect has been observed, Mapolie et al. [7] report nickel complexes based on Periphery-functionalized dendrimers salicylaldime ligand as catalysts for the vinyl polymerization of norbornene. When comparing the effect of dendrimer generation on activity, the second generation catalysts were found to be more active than the first generation catalysts. De la Mata et al. reported the use of titanium-containing carbosilane dendrimers as catalysts for the polymerization of ethylene. Using these metallodendrimers as catalyst precursors, polyethylene with high molecular weight and low polydispersity was produced [8]. The dendritic nature of the catalyst also resulted in a higher degree of crystallinity in the polymers obtained. The Periphery-functionalized dendrimers have their ligand systems, and thus the metal complexes, at the surface of the dendrimer. The transition metals will be directly available for the substrate, in contrast to core-functionalized systems,

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for example, in which the substrate has to penetrate the dendrimer prior to reaction [9]. This accessibility allows reaction rates that are comparable with homogeneous systems. On the other hand, the periphery-functionalized systems contain multiple reaction sites and ligands, which results in extremely high local catalyst and ligand concentrations [10]. This inspireed us to synthesize periphery-functionalized metallodendrimer precatalysts for the polymerization of olefin. Herein we selected amino functionalized dendrimers as a ligand support these dendrimers were modified into metallodendrimes with transition metal ions. This article describes the preparation and characterization of the Ni metallodendrimers, as precatalyst using [PDA-(NH$_2$)$_2$]$_6$ [PDA-(NH$_2$)$_2$]$_{12}$ and abbreviated as C$_1$ (nickel complex of first generation) and C$_2$ (nickel complex of second generation) respectively. The comparison study of the metallodendrimers for ethylene polymerization upon activation with MAO at different pressure.

2 Results and Discussion

A general synthetic route for the first and second generation dendrimeric ligands PAD-(NH$_2$)$_6$ and PAD-(NH$_2$)$_{12}$ is shown in Scheme 1. The PAD-(NH$_2$)$_6$ and PAD-(NH$_2$)$_{12}$ were prepared using ammonium formate and commercial Zinc Powder as a reducing agent from their corresponding PAD-(NO$_2$)$_6$ and PAD-(NO$_2$)$_{12}$ according to the literature

[11]. The yellow powder of 1,3,5 tris(3,5 dinitrophenyl) benzamide [PAD-(NO$_2$)$_6$] was prepared by the reaction of 1,3,5-benzene tricarboxylchloride with 3,5 dinitroaniline in acetone in 1:3 molar ratio in good yield. PAD-(NO$_2$)$_6$ can be purified simply by precipitation in NaHCO$_3$ aqueous in quantitative yield. The formation and isolation of PAD-(NO$_2$)$_6$ were confirmed by IR, elemental analysis, ^{1}H NMR, and ^{13}C NMR spectroscopy as shown Figs. 1 and 2. The IR spectrum of PAD-(NO$_2$)$_6$ showed strong absorption at 1,650 cm^{-1} characteristic to C=O stretching of amide group and terminal nitro groups at 1,588 and 1,290 cm^{-1}. The reduction of PAD-(NO$_2$)$_6$ by employing SnCl$_2$ and concentrated hydrochloric acid successfully afforded the desired 1,3,5 tris(3,5 diaminophenyl) benzamide {PAD-(NH$_2$)$_6$} as a first generated dendrimers. The FTIR spectrum of {PAD-(NH$_2$)$_6$} show a strong peak at 3,410 cm^{-1} due to terminal amino groups [12]. The proton signals at δ 4.36 indicate the presence of NH$_2$ group hydrogen. The 12-amine-terminated second generation polyamide was synthesized when PAD-(NH$_2$)$_6$ react with 3,5-dinitrobenzolyl chloride in acetone under nitrogen atmosphere in 75% yield, then the similar reduction of (5) as used in the synthesis of first generation dendrimers. The structures of PAD-(NH$_2$)$_{12}$ was also characterized by elemental analysis, IR, ^{1}H NMR, and ^{13}C NMR. The ^{1}H NMR spectra of PAD-(NO$_2$)$_{12}$ show proton signals for the internal branches amide and outer branch amide at 10.20 and 10.01 ppm respectively [13]. The terminal amino protons show

Scheme 1 Synthesis of first generation dendrimers and their nickel complexes
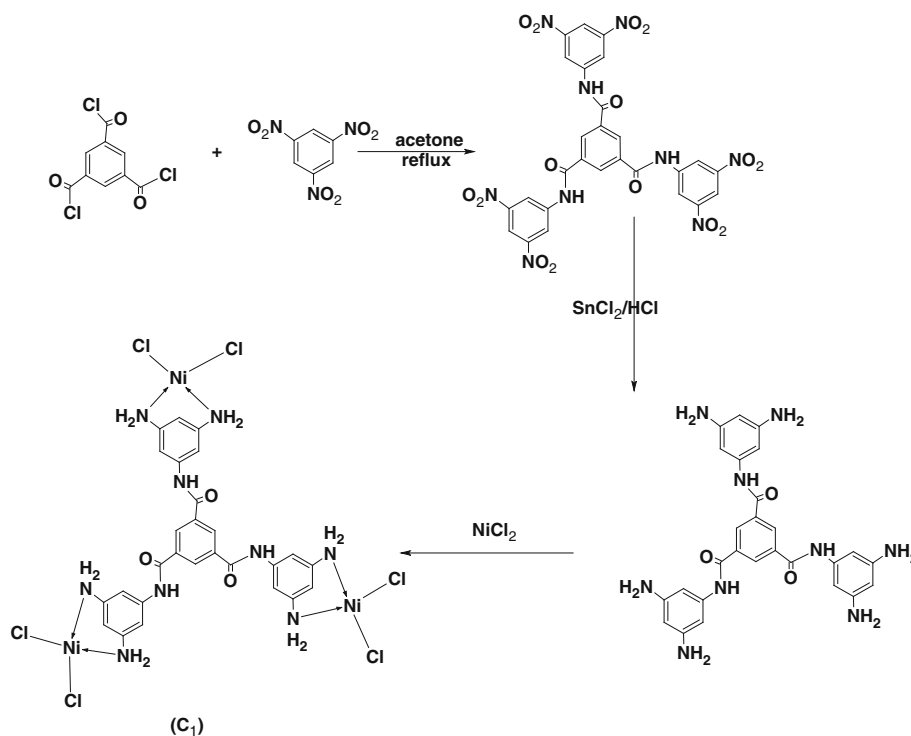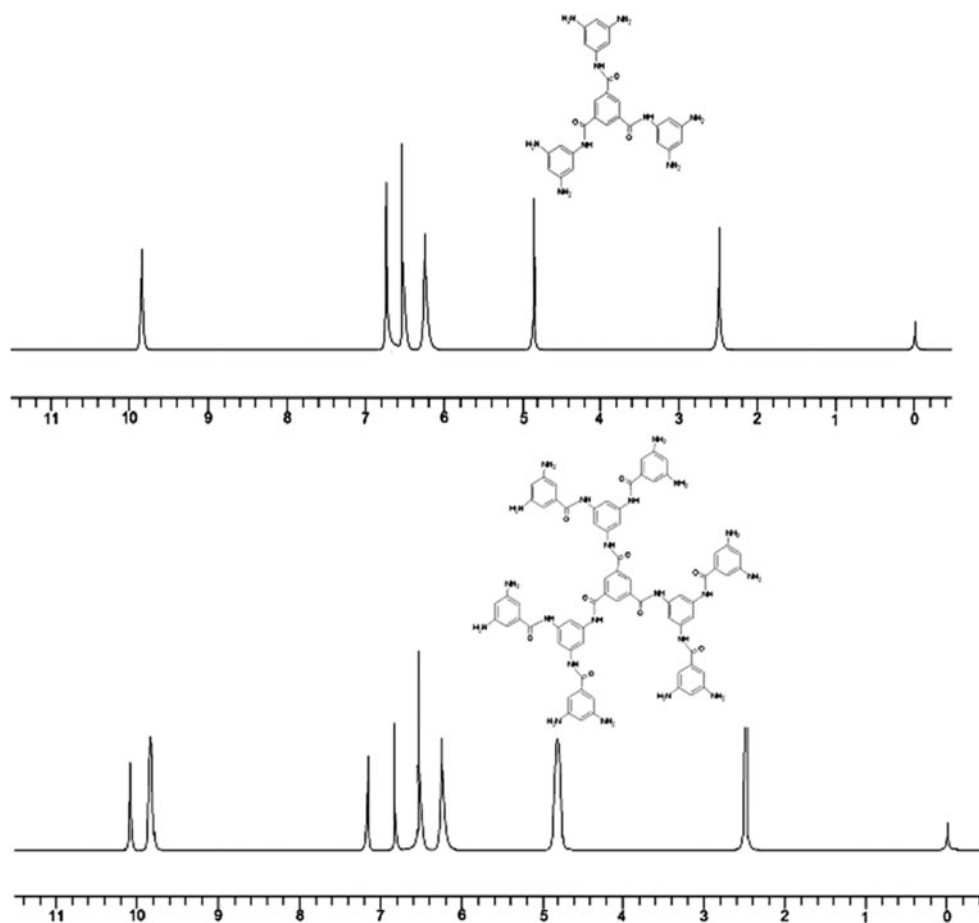

(C$_1$)

Fig. 1 H NMR spectra of first
and second generation
dendrimers

resonance signals at 4.82 ppm. Scheme 2 shows the synthetic route for metallodendrimers of Ni with PAD-$(NH_2)_6$ and PAD-$(NH_2)_{12}$. The metal complexes were synthesized via simple complexation reaction of metal salts with first and second generation dendrimers in ethanol (Scheme 3).

The Nickel complexes were isolated as green colour powder in good yields. The formation of metal complexes was supported by UV–Vis, FTIR, and elemental analysis. The results of elemental analysis revealed that the metal ions attached to the aromatic amine groups via coordination-covalent bonds and containing two chloride ions with each metal ions. In IR spectra of the complexes the NH stretching at 3,405–3,410 cm^{-1} shifted to the lower frequency when compare with their parental dendrimeric ligands [14]. In the metallodendrimers C_1 and C_2, the new strong peak at 530 cm^{-1} is due to the M-N stretch of the bond formed during coordination of Ni via terminal amino group. Although the MALDI-TOF MS measurement of C_1 and C_2 provide a clear spectrum and indicated its formation and isolation as shown in Fig. 3.

Polymerizations of ethylene were conducted at 1 and 5 atmospheric pressure of ethylene in toluene using first generation and second generation metallodendrimers, and methylaluminoxane (MAO) as co-catalyst. We investigated

the effect of Al/Ni ratios which ranged from 50:1 to 2,000:1 molar ratios on the activities of precatalyst first generation C_1 and second generation C_2 for ethylene polymerization at room temperature. The activities of the C_1 and C_2 expressed as turn over frequencies (TOF) and given in Table 1 with 1 atm pressure of ethylene. The oligomeric products were weighed after evaporation of the solvent. C_1 showed optimum catalyst activity at an Al:Ni ratio of 500:1 while C_2 showed optimum activity at an Al:Ni ratio of 1,000:1 when the reaction were carried out with 1 atm pressure of ethylene. As shown in Table 2. the higher polymerization activity was observed at an Al:Ni ratio of 1,000:1 for both catalysts. It is also observed that the ethylene concentration significantly affect the catalytic behaviour of the metallodendrimers. In comparison with the result obtained with 1 atm pressure of ethylene, the distribution of oligomers formed at 5 atm of ethylene pressure shift to higher carbon number of olefins. The oligomers obtained from our C_1 catalyst and C_2 catalyst, were analyzed using gas chromatography (GC). The GC analysis was done on the residues after evaporation of the solvent. This was done after we confirmed that no low boiling point oligomers were present in the reaction medium. From the results it would appear that at low levels of

Fig. 2 C NMR spectra of first and second generation dendrimers
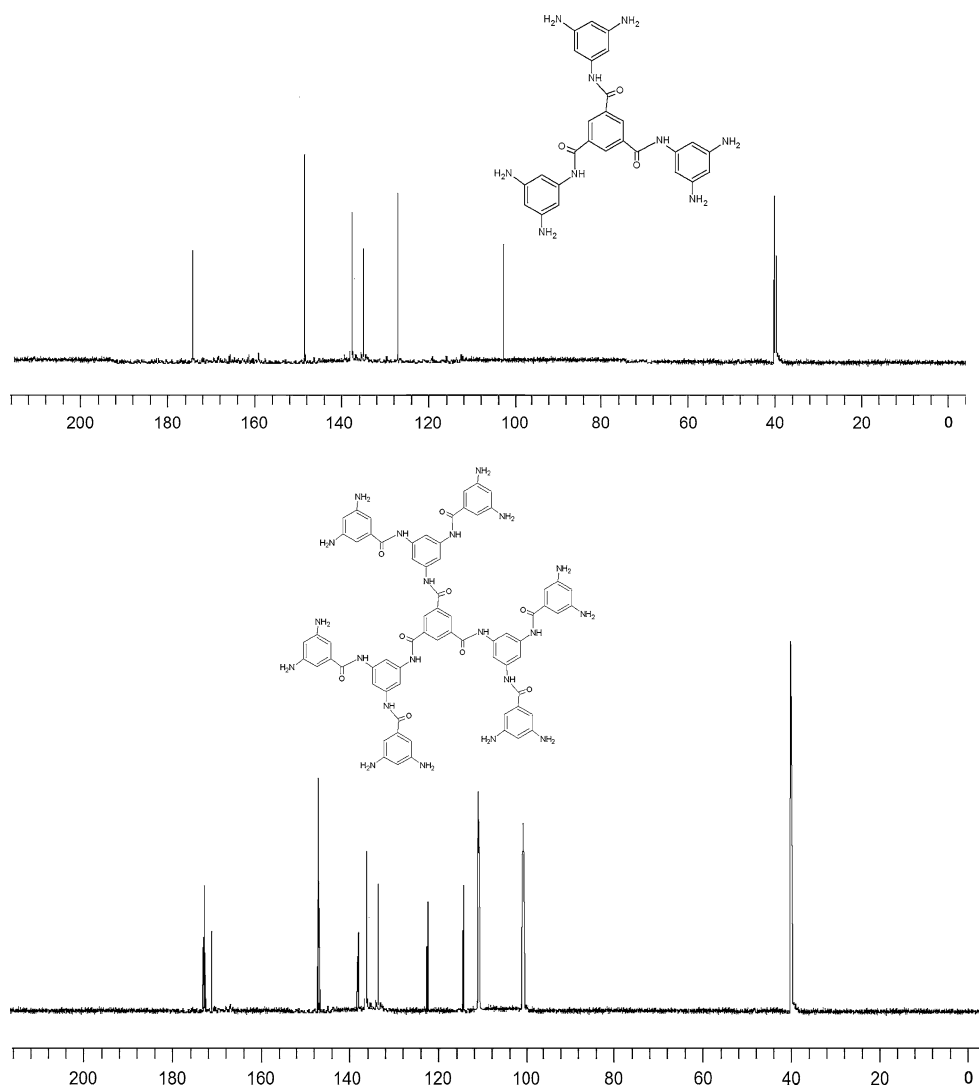
Al, the chain transfer process from the active Ni centre to the Al is much more rapid than chain growth. This results in relatively shorter oligomer chains being formed. At these low Al concentrations, largely C_{10} and C_{12} oligomers were obtained, with C_{10} being more dominant. At very high levels of Al longer chain oligomers were obtained. From our results we observe that our two dendrimer systems give very different types of oligomeric products. Oligomerization using the generation 1 dendrimer complex, C_1 yields short chain oligomers whereas those obtained from the generation 2 dendrimer catalyst, C_2 resulted in longer chain products with molecular weights up to 9.54×10^4 and 9.86×10^4 g/mol at 1 and 5 atm respectively. The overall activity of the generation 2 catalyst is significantly higher than that of the generation 1 catalyst. It should however be noted that the optimum activity for the generation 2 catalyst is obtained at Al concentrations that are twice as high than those used in reactions of the generation 1 catalyst. A

possible reason for this difference in behaviour of the two catalysts in respect of the amount of co-catalyst used could be due to differences in the architecture of the two dendrimeric catalysts [15]. The generation 1 catalyst, C_1 has three amide units within its internal structure, while the generation 2 catalyst, C_2 has six amide units. In both cases these amide, being Lewis base sites, are potential positions for the binding of the Lewis acidic organoaluminium co-catalyst. It is thus thought that the co-catalyst as shown Scheme 2 MAO, first binds to these Lewis basic sites before it becomes involved in the activation of the metal centre. It is well known that N-donor molecules form adducts with Lewis acidic Aluminium complexes [16–18]. Since the C_2 has more N-donor sites it will react with a larger amount of the aluminum alkyl than the C_1. It therefore requires larger amounts of co-catalyst before the optimum activity is reached for the C_2 system.

Scheme 2 Synthesis of second generation dendrimers and their nickel complexes
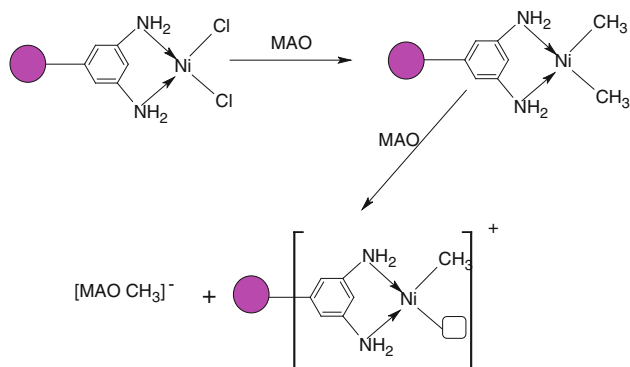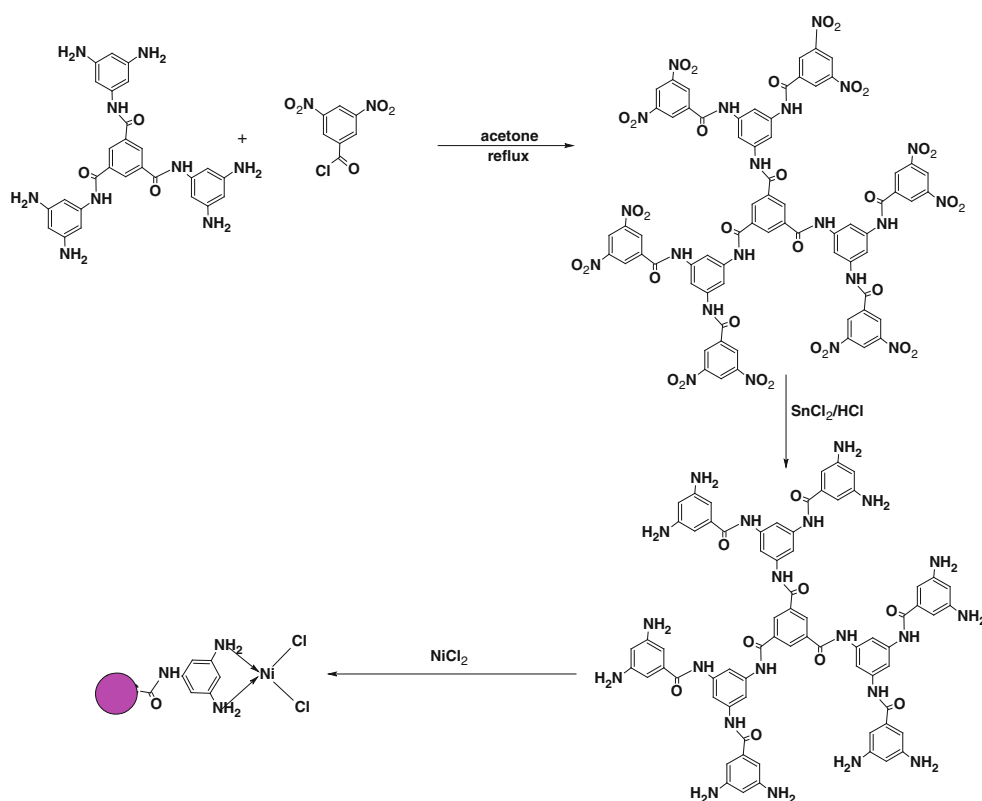
Scheme 3 Mechanism of MOA as a co-catalyst

3 Conclusion

In this study, we have successfully synthesized two new metallodendrimers and used as a catalysts for ethylene oligomerization in the presence of MAO (co-catalyst). The catalytic activities were found to be sensitive to the pressure, co-catalyst to catalyst ratio. The best co-catalyst to catalyst ratio is 1,000:1, at this ratio C_2 exhibit higher activity then C_1, but at 500:1, Al:Ni ratio C_2 show slightly lower catalytic activity than C_1. The type of molecular oligomers formed is also dependent on the nature of the catalyst generation. The C_1 support the formation of short chain oligomers within the C_{10}–C_{20} (50–60%) diesel range,

while C_2 produces higher molecular weight products which were waxy in nature (100%). This behaviour of catalyst C_1 and C_2 can be used in petrochemical industries.

4 Experimental Section

4.1 Materials and Methods

Manipulations of air and moisture sensitive compounds were performed under a nitrogen atmosphere using standard Schlenk techniques. Toluene was refluxed over sodium-benzophenone and distilled under argon prior to use. Methylaluminoxane (MAO, a 1.46 M solution in toluene) and other chemicals were purchased from Sigma-Aldrich. Infrared spectra were recorded on a Perkin Elmer Paragon 1000 PC FT-IR spectrophotometer, using KBr plates. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Varian XR200 spectrometer, using tetramethylsilane as an internal standard. MALDI-TOF Mass spectra were recorded using a KMPACT MALDI mass spectrometer (Shimadzu Kratos) in positive mode. Gas chromatography analysis was carried out on a Varian CP-3800 using a HP PONA column. The yield of oligomers was calculated by referencing to the mass of the solvent on the basis of the prerequisite that the mass of each fraction was approximately proportional to its

Fig. 3 MLDI-TOF-MS of C_1 and C_2
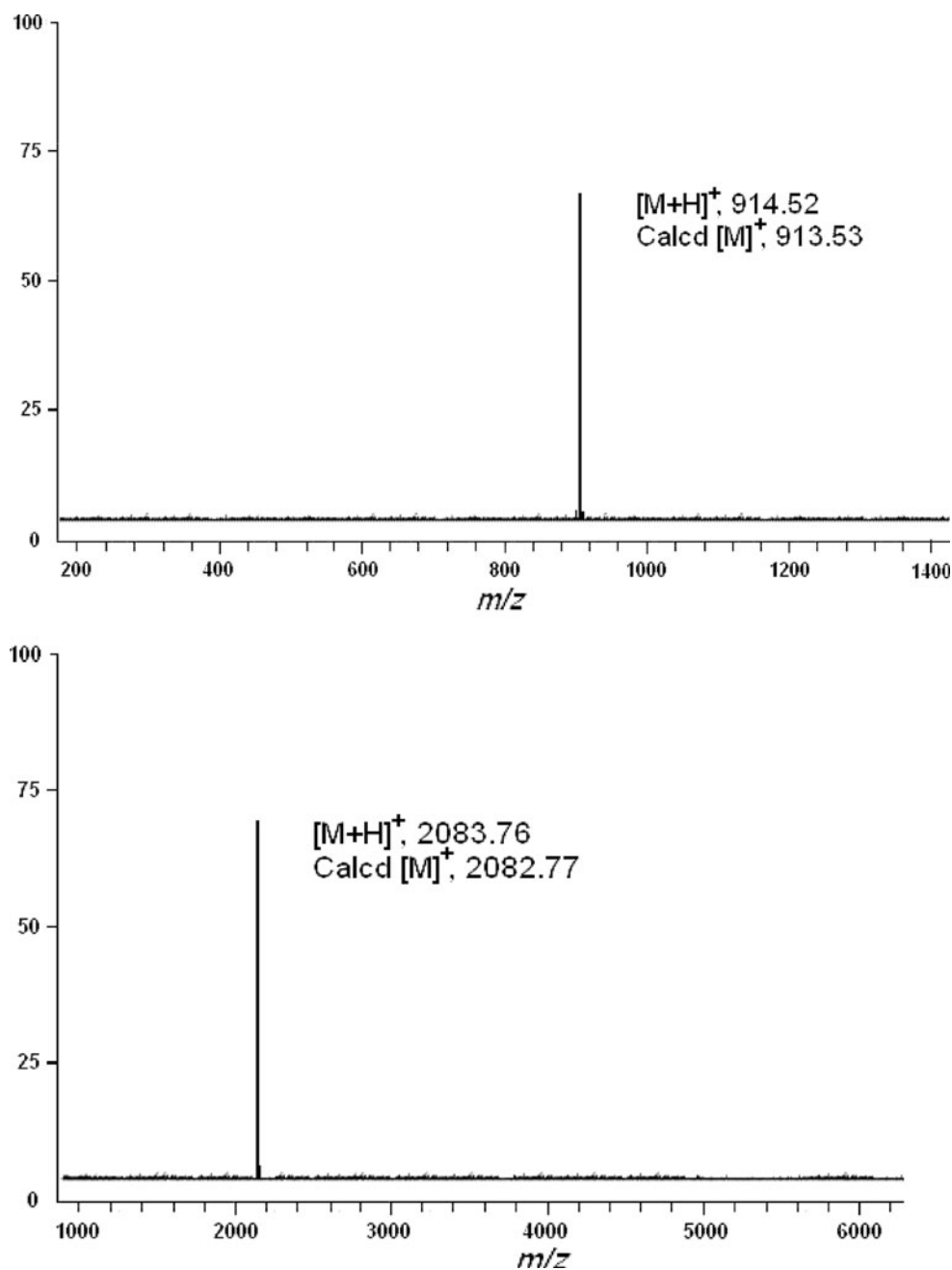
integrated area in the GC trace Dodecane was used as an internal standard. Gel permeation chromatography was performed on a Polymer Laboratories GPC220 instrument using polystyrene standards.

4.2 Synthesis

4.2.1 Synthesis of 1,3,5-Tris(3,5-Dinitrophenyl) Benzamide (1)

1,3,5-Benzene tricarboxylchloride (2.65 g, 0.1 mol) and 3,5-dinitroaniline (2.74 g, 0.3 mol) were mixed in a 250 mL round bottom flask in 100 mL of ethanol. The resulting yellow colour solution was allowed to reflux for 6 h under nitrogen. After the mixture was cooled to room temperature was distilled off under reduced pressure, and the residue was dissolved in hexane resulting precipitate was filtrated and washed off several time. The desired compound [1,3,5-tris(3,5-dinitrophenyl) benzamide)] was obtained as a yellow solid powder in 70% yield after purification by column chromatography (silica gel, 3/1 (v/v) petroleum ether/ethyl acetate). Mp: 190 °C. FTIR (KBr, cm^{-1}): 1110, 1340, 1560, 1588, 1615, 1710, 3265; ^{1}H NMR (400 MHz, DMSO, TMS, δ): 10.46 (s, 3H,

Table 1 Oligomerization of 1 atm of ethylene with C_1–C_2/MAO

Entry	Catalyst	Al/Ni (mol)	Oligomer distribn[a] (%)				Activity[b]	M_w^c ($\times 10^4$) (g/mol)	PDI (M_w/M_n)
			$C_{10}/\Sigma C$	$C_{12}/\Sigma C$	$C_{14}/\Sigma C$	$C_{16+}/\Sigma C$			
1	C_1	50	62	37	1	–	425	–	–
2	C_1	100	58	30	8	4	982	–	–
3	C_1	200	26	50	14	10	1356	–	–
4	C_1	500	2	43	7	48	2450	–	–
5	C_1	1000	–	4	6	90	1845	–	–
6	C_1	2000	–	–	–	100	1536	6.12	1.42
7	C_2	50	–	–	4	16	110	–	–
8	C_2	100	–	–	–	100	460	7.52	1.76
9	C_2	200	–	–	–	100	812	7.78	1.94
10	C_2	500	–	–	–	100	1427	8.24	1.49
11	C_2	1000	–	–	–	100	3250	9.54	1.65
12	C_2	2000	–	–	–	100	2600	7.65	1.47

General condition: 5 μmol of catalyst; 30 min; room temperature, 30 mL of toluene

[a] Determined by GC; ΣC signifies the total amounts of oligomers

[b] TOF: 103 g of polyethylene produced per mol of Ni per hour per bar

[c] Determined by GPC

Table 2 Oligomerization of 5 atm of ethylene with C_1–C_2/MAO

Entry	Catalyst	Al/Ni (mol)	Oligomer distribn[a] (%)				Activity[b]	M_w^c ($\times 10^4$) (g/mol)	PDI (M_w/M_n)
			$C_{10}/\Sigma C$	$C_{12}/\Sigma C$	$C_{14}/\Sigma C$	$C_{16+}/\Sigma C$			
1	C_1	50	59	32	9	–	420	–	–
2	C_1	100	53	21	14	12	840	–	–
3	C_1	200	21	30	19	20	1295	–	–
4	C_1	500	–	10	53	37	1920	–	–
5	C_1	1000	–	–	–	100	2360	6.86	1.46
6	C_1	2000	–	–	–	100	1820	7.42	1.56
7	C_2	50	–	–	–	100	230	7.86	1.84
8	C_2	100	–	–	–	100	500	8.12	1.88
9	C_2	200	–	–	–	100	960	8.65	1.56
10	C_2	500	–	–	–	100	1590	9.22	1.64
11	C_2	1000	–	–	–	100	3280	9.86	1.76
12	C_2	2000	–	–	–	100	2430	8.02	1.52

General condition: 5 μmol of catalyst; 30 min; room temperature, 30 mL of toluene

[a] Determined by GC; ΣC signifies the total amounts of oligomers

[b] TOF: 103 g of polyethylene produced per mol of Ni per hour per bar

[c] Determined by GPC

N$\underline{H}$CO), 7.12 (s, 3H, Ar$\underline{H}$), 6.96 (s, 3H, Ar$\underline{H}$), 6.42 (s, 6H, Ar$\underline{H}$). Anal. Calcd for $C_{27}H_{15}N_9O_{15}$ (705.46): C, 45.97; H, 2.14; N, 17.87. Found: C, 45.95; H, 2.13; N, 17.89.

4.2.2 Synthesis of First-Generation Dendritic Ligand (2)

To a solution of 1 (3.52 g, 50 mmol in 30 mL of acetone) and Sn (7.64 g), conc. HCl (60 mL) was drop wised added and refluxed for 30 min. Cool the flask and add a solution of sodium hydroxide in 100 cm^3 of water to re-dissolve the initial precipitate. Add 3 g of powdered sodium chloride to the mixture, shake to dissolve, and then transfer the liquid to a separating funnel and 10 mL of chloroform. The separated organic mixture was washed off several times with water. The solvent was evaporated at reduced pressure, and the desired compound 2 was obtained as a brown

colours powder in 54% yield after purification by column chromatography (silica gel, 3/1 petroleum ether/ethyl acetate). Mp: 245 °C, FTIR (KBr, cm^{-1}): 1511, 1556, 1596, 1622, 1680, 3409. ^{1}H NMR (400 MHz, DMSO, TMS, δ): 9.84(s, 3H, N$\underline{H}$–CO), 6.81 (s, 3H, Ar$\underline{H}$), 6.52 (s, 6H, Ar$\underline{H}$), 6.21 (s, 3H, Ar$\underline{H}$) and 4.83 (s, 12H, Ar–N$\underline{H}_2$); ^{13}C NMR (100 MHz, DMSO, TMS): 102.3, 128.4, 134.9, 136.8, and 174.8 ppm. ESI-MS (m/z): 526.23 (M + H$^+$). Anal. Calcd for $C_{27}H_{27}N_9O_3$ (525.56): C, 61.70; H, 5.18; N, 23.99. Found: C, 61.71; H, 5.17; N, 23.98.

4.2.3 Synthesis of Second Generation Nitro Group Terminated Dendrimers (3)

To a solution of 2 (5.26 g, 10 mmol) in chloroform (100 mL) was added 1,3-dinitrobenzoyl chloride (13.99 g, 60 mmol) and the mixture was refluxed under nitrogen, with stirring at 70 °C for 5 h resulting yellow colour mixture found. The process of reaction was monitored by TLC using CH_2Cl_2 and hexane as eluent. After completion of the reaction the solvent was removed under reduced pressure and washed off several time using water, and obtained as a yellow brown powder in 60% yield. Mp: 275 °C. FTIR (KBr, cm^{-1}): 1115, 1342, 1560, 1558, 1588, 1650, 1720, 3245; ^{1}H NMR (400 MHz, DMSO, TMS, δ): 9.81 (s, 3H, N$\underline{H}$–CO), 9.76 (s, 6H, N$\underline{H}$–CO), 7.27 (s, 9H, Ar$\underline{H}$), 6.84 (s, 3H, Ar$\underline{H}$), 6.62 (s, 6H, Ar$\underline{H}$), and 6.25 (s, 12H, Ar$\underline{H}$). ^{13}C NMR (100 MHz, DMSO, TMS): 104.2, 112.5, 114.2, 129.8, 133.3, 135.4, 137.2, 145.2, 171.8 and 174.1 ppm. Anal. Calcd for $C_{69}H_{39}N_{21}O_{33}$ (1690.17): C, 62.29; H, 4.77; N, 22.11. Found: C, 62.30; H, 4.78; N, 22.10.

4.2.4 Synthesis of Second Generation Dendritic Ligand (4)

Using the same procedure as for the synthesis of 3 was obtained as a yellow powder in 52% yield as a brown powder. Mp: 348 °C. FTIR (KBr, cm^{-1}): 1114, 1340, 1562, 1560, 1586, 1652, 1721, 3265; ^{1}H NMR (400 MHz, DMSO, TMS, δ): 4.82 (s, 48H, N$\underline{H}_2$), 10.10 (s, 3H, N$\underline{H}$–CO), 9.82 (s, 6H, N$\underline{H}$–CO), 7.18 (s, 9H, Ar$\underline{H}$), 6.82 (s, 3H, Ar$\underline{H}$), 6.48 (s, 6H, Ar$\underline{H}$), and 6.21 (s, 12H, Ar$\underline{H}$). ^{13}C NMR (100 MHz, DMSO, TMS): 103.2, 110.8, 115.4, 123.8, 134.2, 136.0, 138.4, 147.0, 170.6 and 173.2 ppm. Anal. Calcd for $C_{69}H_{39}N_{21}O_9$ (1330.51): C, 49.03; H, 2.33; N, 17.40. Found: C, 49.01; H, 2.24; N, 17.42.

4.2.5 Synthesis of Generation 1 Dendritic Nickel Complex (C₁)

To the 1,3,5-tris(3,5-diaminophenyl) benzamide (1.31 g, 5 mmol) in ethanol (25 mL) in a round bottom flask was added Nickel chloride hexahydrate (3.80 g, 15 mmol) and

the reaction mixture was allowed to stir under reflux for 24 h forming a green precipitate. The precipitate was filtered off by vacuum filtration and washed extensively with ethanol to afford C_1 as a green solid in 80% yield. Mp: 245 °C, FTIR (KBr, cm^{-1}): 1515, 1550, 1584, 1620, 1680, 3374. Anal. Calcd for $C_{27}H_{27}N_9O_3Ni_3Cl_6$ (914.35): C, 35.47; H, 2.98; N, 13.79; Cl, 23.26; Ni, 19.26. Found: C, 35.47; H, 2.98; N, 13.79, Cl, 23.26, Ni, 19.26.

4.2.6 Synthesis of Generation 2 Dendritic Nickel Complex (C₂)

To a solution of the PAD-(NH$_2$)$_{12}$ dendrimeric ligand (6.65 g, 5 mmol) in ethanol (10 mL) was added nickel chloride hexahydrate (7.60 g, 30 mmol) and the mixture stirred under reflux for 24 h. The solvent was evaporated via rotary evaporation to give a green residue. The residue was then dissolved in dichloromethane (15 mL) and the solution was filtered by gravity and dried to afford complex C_2 as a green solid in 76% yield. Mp: 348 °C. FTIR (KBr, cm^{-1}): 1110, 1342, 1560, 1556, 1582, 1648, 1712, 3260; $C_{69}H_{39}N_{21}O_9Ni_6Cl_{12}$ (2083.77): C, 39.77; H, 1.89; N, 14.12; Cl, 20.42; Ni, 16.90. Found: C, 39.78; H, 1.88; N, 14.14, Cl, 20.43, Ni, 16.91.

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