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Polymeric catalyst with polymerization-enhanced Lewis acidity for CO_2-based copolymers

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ABSTRACT

Ring-opening copolymerization of CO_2 and epoxides is a promising way to manufacture high value-added materials. Despite a variety of catalyst systems have been reported, the reaction is still limited by low activity and polymer selectivity. Herein, a strategy of polymerization-enhanced Lewis acidity is reported to construct a series of highly efficient polymeric aluminum porphyrin catalysts (PAPCs). The characterization of the coordination equilibrium constant (K_{eq}) showed significantly enhanced Lewis acidity of PAPC ($K_{eq} = 18.2$ L/mol) compared to the monomeric counterpart ($K_{eq} = 6.4$ L/mol), accompanied with increased turnover frequency (TOF) from 136 h^{-1} to 5500 h^{-1}. Through detailed regulation of Lewis acidity, the highly Lewis acidic PAPC-OTs displayed a record high TOF of 30,200 h^{-1} with polymer selectivity of up to 99%.

Carbon dioxide (CO_2), the main component of greenhouse gasses, can be converted into high-value-added polymers through the ring-opening copolymerization (ROCOP) reaction with epoxides. This ROCOP reaction not only reduces the environmental burden caused by CO_2, but also lessens the reliance of polymer materials on petroleum supplies, gaining great attention since its first discovery by Inoue *et al.* [1]. Despite of low catalytic activity (turnover frequency, TOF of only 0.12 h^{-1}), this reaction opens new avenues for the designing of efficient catalysts. With the continuous investigation over the past decades, dramatic development has been witnessed and some highly efficient catalysts have been reported, typical examples being the β-diiminate zinc (BDI-Zn) catalyst [2–4], porphyrin systems [5–11], and salen systems [12–15]. It is noticeable that bifunctional salenCo was extraordinarily effective for the copolymerization of CO_2/propylene oxide (PO), setting a record TOF of 26,000 h^{-1} [13]. Additionally, Williams *et al.* established a series of Robson-type bimetallic complexes which displayed outstanding activity for CO_2/cyclohexene oxide (CHO) copolymerization even under 1 atm pressure [16–18]. Pioneered by Gnanou and Feng [19], copolymerization under metal-free conditions represented another great advancement [20–24]. Notwithstanding the great progress, the activity of current catalysts, regrettfully, is still far from satisfactory, displaying 2–3 orders of magnitude lower than the Ziegler-Natta catalysts in olefin polymerization [25]. Hence, designing more efficient catalysts has always been a big pursuit, as this means lower catalyst cost, by-product separation cost, and time cost in practice.

To better design catalysts, an in-depth understanding of the catalytic reaction mechanism is indispensable. It is generally accepted that the rate-determining step is the ring-opening of epoxide during the ROCOP process [26,27]. Therefore, enhancing the Lewis acidity of the catalyst center that facilitating the coordination and ring-opening of epoxides has been regarded as a valid way to boost catalytic efficiency [28,29]. Among all the methods for the regulation of Lewis acidity, variation of the catalytically active metal center or the ligand environment (*e.g.*, ligand type, substituent, axial group) has received considerable attention. Ema *et al.* systematically altered the above elements based on the bifunctional porphyrin ligand, achieving a TOF of 10,000 h^{-1} for CO_2/CHO copolymerization [8]. Williams and coworkers reported a MgCo binuclear catalyst by regulating metal centers, giving a TOF of 12,460 h^{-1} with polymer selectivity > 99% [30]. Lu *et al.* delved into the trinuclear salenCo system modulated by axial groups, where catalyst with strong Lewis acidity showed a high TOF (2100 h^{-1}) and polymer selectivity (99%), much better than that of the weak counterpart (TOF of 80 h^{-1} and polymer selectivity of 51%) [31]. Besides, this Lewis acidity regulation strategy is also widely applied in many other fields such as hydrogenations [32,33], C–H

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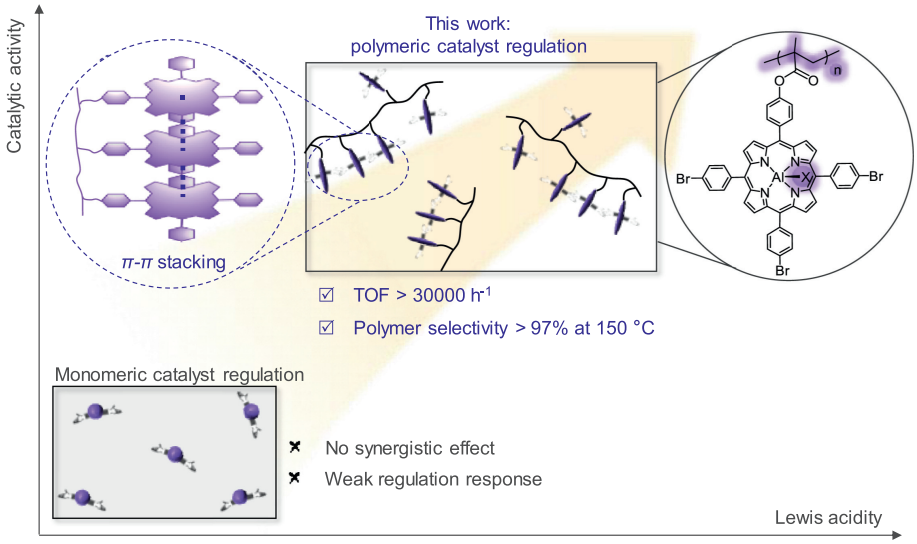


Scheme 1. Schematic illustration of polymerization-enhanced Lewis acidity for boosted catalytic activity in comparison with monomeric system.

borylations [34], aminations [35], and vinyl monomer polymerizations [36,37]. Despite these achievements, past works on Lewis acidity regulation was mostly based on trial and error, after which leading catalysts were engineered by tuning above factors. Therefore, it is essential to develop a designed way to amplify the Lewis acidity for improved activity.

In this study, we propose a strategy of polymerization-enhanced Lewis acidity by polymerizing the monomeric catalysts into polymeric ones for precise boosting the catalytic activity of CO_2/PO copolymerization (Scheme 1). As a proof-of-concept, the as-prepared polymeric aluminum porphyrin catalyst (PAPC) displayed stronger Lewis acidity (coordination equilibrium constant, $K_{eq} = 18.2$ L/mol) than the corresponding monomeric counterpart ($K_{eq} = 6.4$ L/mol), along with the boosted catalytic reactivity from TOF = 136 h^{-1} to 5500 h^{-1}, demonstrating the feasibility of the present strategy. To further regulate the Lewis acidity, facile yet efficient axial group engineering of PAPC was adopted, affording a significantly enhanced TOF from 2700 h^{-1} to 10,600 h^{-1} with an increase of K_{eq} from 14.3 L/mol to 48.7 L/mol. Notably, PAPC-OTs with *p*-tosylate group displayed a record high TOF of 30,200 h^{-1} with high polymer selectivity of 99%.

The key element of polymerization-enhanced Lewis acidity strategy lied in the construction of polymeric catalysts that showed improved activity for CO_2/PO copolymerization in comparison with monomeric counterparts. Aluminum porphyrin catalyst with a large 18 π-electron conjugated structure displayed high catalytic performance, owing to their strong intermolecular synergistic effects [38]. Therefore, as shown in Scheme S1 (Supporting information), PAPC with pendent aluminum porphyrin units was designed and synthesized. On the basis of spatial confinement of polymer main chain and synergistic effects of porphyrin units, an accurate enhancement of Lewis acidity of polymeric catalyst can be achieved. To further regulate the Lewis acidity of PAPC, ligand substitution engineering including electronic or steric effect is a conventional way. However, owing to the large conjugated structure of the porphyrin ligand, it is hard to have a direct impact on the Lewis acidity of metal center by tuning substituent groups. The axial groups, directly connected to metal center, represent a valid way for tuning the electrical properties. Consequently, we chose six axial groups with distinct electron-withdrawing abilities, following the order ethyl < acetate < chloride < nitrate < *p*-tosylate < perchlorate, and prepared the corresponding catalysts PAPC-X (X = Et, OAc, Cl, NO_3, OTs, ClO_4). As a control, corresponding monomeric aluminum porphyrin catalysts (MAPCs) were prepared. The chemical structures of the polymeric catalysts, monomeric catalysts and the associated intermediates were confirmed by ^{1}H NMR spectroscopy, matrix-assisted laser desorption ionization time-of-flight mass spectrometry (MALDI-TOF-MS), and gel permeation chromatography (GPC) (Figs. S1-S14 in Supporting information). The characterization results revealed the well-defined polymer structure of PAPC with an average polymerization degree of about 9.

A large number of efforts have been dedicated to measure the strength of Lewis acids, including spectroscopic methods [39,40] and computational methods [41,42]. Since the capacity to absorb electrons is the essence of Lewis acidity, Chisholm *et al.* reported a facile way of determining Lewis acidity based on the binding of the azide ion to porphyrin catalysts by an IR study [5]. As illustrated in Figs. 1a and b, from the IR spectra of coexistence of free (2075 cm^{-1}) and bound (2125 cm^{-1}) azide signals in PAPC/$PPNN_3$ mixtures, the K_{eq} of PAPC can be calculated from equation based on the integral areas of the bound azide compared to the free azide. The PAPC displayed significantly enhanced Lewis acidity than the monomeric catalysts (Fig. 1c and Table S1 in Supporting information). For example, the K_{eq} of PAPC-Et (14.3 L/mol) was much higher than that of MAPC-Et (2.3 L/mol), indicating the validity of the polymerization-enhanced Lewis acidity strategy. Further tuning of axial groups, more electrophilic group such as ClO_4 enabled a stronger Lewis acidity for both PAPC and MAPC. With an increase of electron withdrawing ability in axial group X from Et, OAc, Cl, NO_3, OTs and then to ClO_4, PAPC-X possessed a progressively enhanced K_{eq} from 14.3, 16.8, 18.2, 25.6, 38.0 and then to 48.7 L/mol, respectively, higher than the MAPC-X counterparts (2.3, 4.5, 6.4, 8.4, 8.8, 10.4 L/mol, respectively) (Table S1, Figs. S15 and S16 in Supporting information). These results demonstrated the amplification effect of polymeric catalysts *versus* monomeric systems.

To better understand the origin of polymerization-enhanced Lewis acidity strategy, we conducted the ^{1}H NMR and UV–vis studies on both polymeric and monomeric porphyrin ligands. As shown in Fig. 1d, the polymeric porphyrin ligand exhibited a significant blue shift in the S-band (409 nm) compared to that of the monomeric porphyrin ligand (417 nm). Meanwhile, an upfield shift of proton signal for ring protons (−3.6 *vs.* −2.8 ppm) in polymeric porphyrin ligand *versus* monomeric system was observed (Fig. 1e). These results indicated the tendency of H aggregation of porphyrin units within the polymeric ligand, featuring a face-to-face stacking

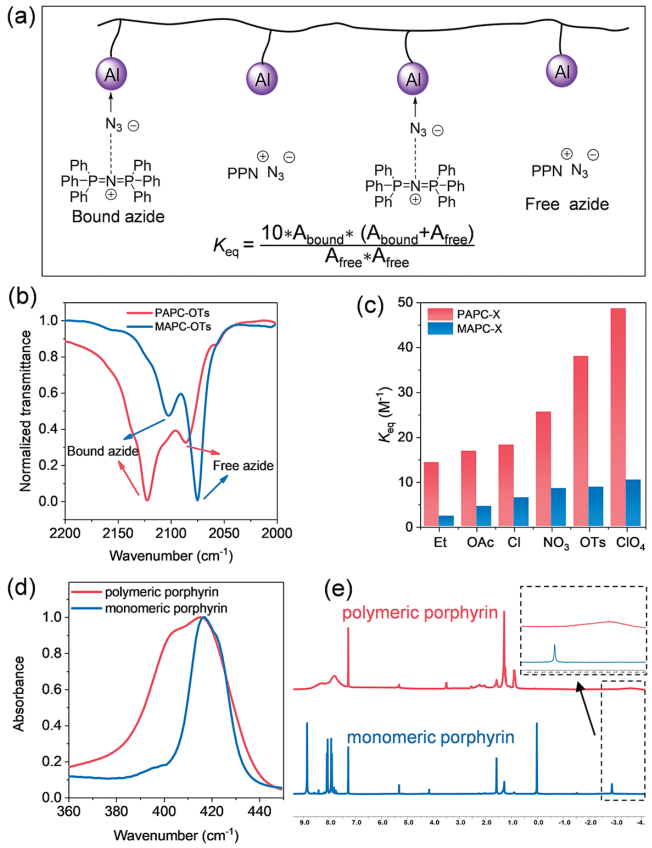


Fig. 1. (a) Schematic illustration of the coordination equilibrium between PAPC and $PPNN_3$. (b) FT-IR spectra of equal molar ratios of $PPNN_3$ to PAPC-OTs and MAPC-OTs, respectively, in dichloromethane solution. (c) The comparison of K_{eq} value for PAPC-X and MAPC-X. (d) UV–vis spectra of polymeric (red) and monomeric (blue) porphyrin ligand. (e) Stacked ^1H NMR spectra of polymeric (red) and monomeric (blue) porphyrin ligand.

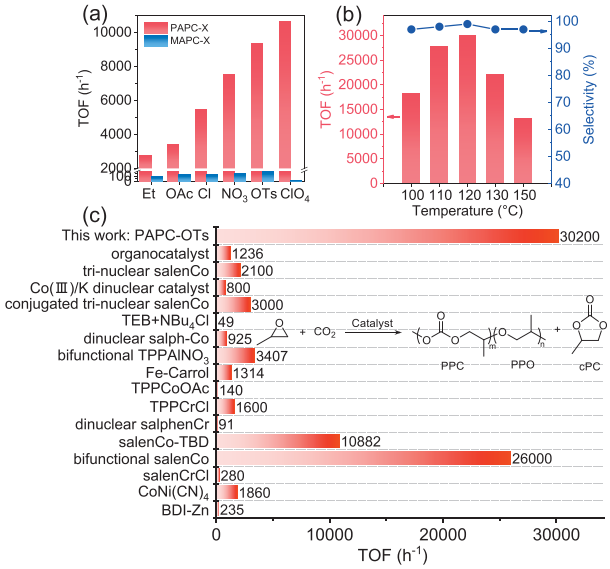


Fig. 2. (a) Catalytic performance comparison of PAPC-X and MAPC-X towards CO_2/PO copolymerization. (b) TOF of PAPC-OTs under different temperatures. (c) Comparison of PAPC-OTs with reported catalyst systems for CO_2/PO copolymerization. The data represents the highest TOF values for each catalyst, and the corresponding structures of catalysts are shown in Fig. S19 (Supporting information).

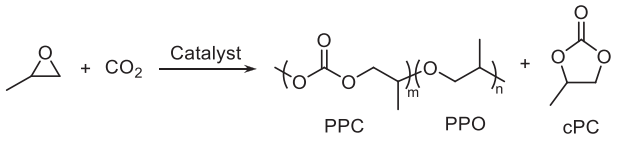
mode [43,44]. We reasoned that the H aggregation of porphyrin units in PAPC resulted in the enhancement of the Lewis acidity, possibly owing to the delocalization of π electron in porphyrin.

To investigate the relationship between Lewis acidity and catalytic performance, the as-prepared PAPCs were utilized for the ROCOP of PO and CO_2 with corresponding MAPCs as controls. The polymerizations were carried out with a catalyst loading of 0.005 mol% at 70 °C under 4 MPa CO_2 pressure in the presence of $(Ph_3P{=}N{=}PPh_3)^+Cl^-$ (PPNCl) cocatalyst. As displayed in Table 1 and Fig. 2a, the polymeric catalyst PAPC-Et displayed a high TOF of 2700 h^{-1}, outperforming the monomeric counterpart MAPC-Et (TOF = 92 h^{-1}). This result demonstrated that the polymerization-enhanced Lewis acidity strategy greatly boosted the catalytic activity. With an increase of electron withdrawing ability in axial group from Et, OAc, Cl, NO_3, OTs and then to ClO_4, the TOF of PAPC-X increased steadily in the order of 2700, 3400, 5500, 7500, 9300 and 10,600 h^{-1}, significantly higher than the corresponding monomeric MAPC-X series (92, 132, 136, 154, 193 and 17 h^{-1}), again indicating the positive effect of Lewis acidity on catalytic activity. Meanwhile, PAPC with strong electron-withdrawing axial group such as Cl, NO_3, OTs and ClO_4 displayed high polymer selectivity (>99%) without the detection of cyclic propylene carbonate (cPC) byproduct (Fig. S17 in Supporting information). Meanwhile, the end-chain structure of the resulting copolymers was confirmed by MALDI-TOF-MS (Fig. S18 in Supporting information). It is noticeable that PAPC-ClO_4 showed a high TOF of 10,600 h^{-1}, delivering nearly a 625-fold improvement over the MAPC-ClO_4 (17 h^{-1}) and a 3.9-fold increase than PAPC-Et (2700 h^{-1}). Unlike the positive correlation of TOF with Lewis acidity in polymeric catalyst series, the TOF of MAPC-ClO_4 with relatively high Lewis acidity was inferior to other monomeric catalysts, owing to the weak nucleophilic ability of ClO_4 group. While for polymeric counterpart PAPC-ClO_4, we reasoned that the enhanced Lewis acidity facilitated the ring opening of epoxide even for the weakly nucleophilic ClO_4 group. The effect of CO_2 pressure was investigated as shown in Table S2 (Supporting information). The catalytic activity of PAPCs increased under lower pressure (from 5500 h^{-1} at 4 MPa to 10,200 h^{-1} at 0.1 MPa) with high selectivity, as bimetallic enchainment is more favourable for homopolymerization in comparison with PO/CO_2 copolymerization [45]. Additionally, the resulting polymers obtained by polymeric catalysts showed a carbonate unit (CU) content of about 40% except for PAPC-ClO_4 (CU < 5%), which is more suitable as a highly active catalyst for polyether synthesis (Table S3 in Supporting information). In contrast, PAPC-OTs displayed both suitable CU and high activity, which is further utilized for optimization of polymerization conditions.

To further boost the catalytic activity, increasing reaction temperature is a valid way. However, most catalysts suffered from low activity and selectivity at high temperature, due to the structural instability [12,46,47]. In our study, the TOF of PAPC-OTs was nearly doubled from 9300 h^{-1} to 18,300 h^{-1} with an increase of temperature from 70 °C to 100 °C (Table 1, entries 5 and 13). Further increase of temperature to 150 °C, the TOF increased first and then decreased, with the maximum value reaching 30,200 h^{-1} at 120 °C companied by a high polymer selectivity of 99% (Fig. 2b and Table 1, entry 15). With decline of activity at temperature above 120 °C, probably due to partial deactivation of the catalyst, the polymer selectivity still remained above 97% (Table 1, entries 16 and 17), which is challenging for other catalysts [6,12,48]. As summarized in Fig. 2c, PAPC-OTs showed unprecedented activity in comparison to the other reported catalysts, which is even 15% higher than the record bifunctional salenCo (TOF of 26,000 h^{-1}) [13]. All these results proved that polymerization-enhanced Lewis acidity is efficient for the enhancement of catalytic performance (activity and polymer selectivity) of CO_2/PO copolymerization.

A consideration of possible mechanistic issues brings an understanding of the origin of high catalytic activity and selectivity

Table 1
Catalytic performance comparison of PAPC and MAPC towards CO_2/PO copolymerization.[a]



Entry	Catalyst	[PO]/[Al] [b]	T (°C)	t (h)	TOF (h^{-1}) [c]	Selectivity (%) [d]	CU (%)[e]	M_n (kg/mol) [f]	PDI [f]
1	PAPC-Et	20,000	70	3	2700	95	47	43.0	1.17
2	PAPC-OAc	20,000	70	0.5	3400	94	37	18.4	1.69
3	PAPC-Cl	20,000	70	0.5	5500	>99	40	14.9	1.45
4	PAPC-NO_3	20,000	70	0.5	7500	>99	40	20.0	1.61
5	PAPC-OTs	20,000	70	0.5	9300	>99	37	19.1	1.87
6	PAPC-ClO_4	20,000	70	0.5	10,600	>99	3.6	13.3	1.78
7	MAPC-Et	5000	70	12	92	93	96	7.2	1.80
8	MAPC-OAc	5000	70	12	132	69	97	12.9	1.26
9	MAPC-Cl	5000	70	12	136	69	97	12.9	1.26
10	MAPC-NO_3	5000	70	12	154	77	98	10.5	1.37
11	MAPC-OTs	5000	70	12	193	93	92	19.4	1.47
12	MAPC-ClO_4	5000	70	12	17	>99	<1	1.9	1.08
13	PAPC-OTs	20,000	100	0.17	18,300	97	34	10.1	1.53
14	PAPC-OTs	20,000	110	0.17	27,700	98	34	8.7	2.08
15	PAPC-OTs	30,000	120	0.25	30,200	99	37	12.9	1.81
16	PAPC-OTs	30,000	130	0.25	22,100	97	33	7.5	1.41
17	PAPC-OTs	30,000	150	0.20	13,200	97	24	6.1	1.92

[a] The polymerization reaction was carried out in neat PO (3 mL) at 4 MPa of CO_2 pressure with [Al]/[PPNCl] molar ratio of 1:1.
[b] Molar ratio of PO to Al centers in polymeric or monomeric catalysts.
[c] Turnover frequency, calculated by (mol PO to products)/(mol Al × time).
[d] Molar selectivity for polymer over cPC.
[e] Carbonate unit content in polymer, determined by ^{1}H NMR analysis.
[f] Determined by gel permeation chromatography in CH_2Cl_2 at 35 °C, calibrated with polystyrene standards.

in PAPC. In the initial stage of ROCOP reaction, compared with monomeric catalyst, the enhanced Lewis acidity in PAPC derived from the intermolecular interactions of porphyrin units in confined polymer backbone not only facilitated the coordination and activation of epoxide for enhanced activity, but also stabilized the growing polymer chains for improved polymer selectivity by restricting backbiting side reactions. Interestingly, unlike conventional catalysts in which the axial group directly attacked epoxide and then attached to the polymer chain end, the electron-withdrawing axial group in PAPC preferred to partially participated in the initiation process while leaving free axial group on the metal center that can continuously regulate the Lewis acidity of the catalyst during the polymerization reaction (Scheme S2 in Supporting information), which was well demonstrated by the activity difference between PAPC-Cl and PAPC-ClO_4 in the middle period of polymerization by *in-situ* IR (Table S4 and Fig. S20 in Supporting information). Collectively, the axial groups shuttle among the multiple aluminum porphyrin centers in the PAPC backbone may play an overall role in improving the ultra-high activity and selectivity at high temperatures.

In summary, with the strategy of polymerization-enhanced Lewis acidity, a series of highly active and selective polymeric catalysts for the CO_2/PO copolymerization were prepared. Verified by the coordination equilibrium constant, we found that the Lewis acidity of PAPCs were greatly enhanced compared to MAPCs, due to the H aggregation of porphyrin units. Moreover, the enhanced Lewis acidity resulted in greatly boosted activity (TOF value increased up to 625-fold) and polymer selectivity (as high as 99%). Among polymeric catalysts, the highly Lewis acidic PAPC-OTs provided an unprecedented TOF of 30,200 h^{-1}, which is the highest record achieved to date for the CO_2/PO copolymerization. And it can endure a temperature as high as 150 °C with a polymer selectivity >97%. With targeted regulation of Lewis acidity, this work provides a new design strategy for highly active and selective catalyst.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2023.108630.

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