Multicatalytic Transformation of (Meth)acrylic Acids: a One-Pot Approach to Biobased Poly(meth)acrylates

Hugo Fouilloux, Wei Qiang, Carine Robert, Vincent Placet, and Christophe M. Thomas*

Abstract: Shifting from petrochemical feedstocks to renewable resources can address some of the environmental issues associated with petrochemical extraction and make plastics production sustainable. Therefore, there is a growing interest in selective methods for transforming abundant renewable feedstocks into monomers suitable for polymer production. Reported herein are one-pot catalytic systems, that are active, productive, and selective under mild conditions for the synthesis of copolymers from renewable materials. Each system allows for anhydride formation, alcohol acylation and/or acid esterification, as well as polymerization of the formed (meth)acrylates, providing direct access to a new library of unique poly(meth)acrylates.

Introduction

Cheap, light and versatile plastics are the dominant materials of our modern economy. The vast majority of these commodity materials are obtained from fossil fuels. In order to remedy some of the environmental challenges associated with petrochemical extraction, an alternative to fossil feedstocks involves using chemicals from renewable resources. In particular, the development of new methods for transforming biomass into resources suitable for polymer production is a critical hurdle along the path to a more sustainable chemical economy. The main challenge is then to design efficient and selective transformations of abundant, renewable, low-cost raw materials into innovative polymeric products. Catalysis is as an important tool to support a more sustainable plastics production and in this case should ideally be efficient, convenient, and versatile, using common reagents. In this regard, one-pot catalytic transformations have significant advantages over conventional multi-step syntheses such as time- and cost-savings, waste reduction and energy consumption. These synthetic schemes, which proceed through two or more consecutive catalytic steps, may serve as a versatile method in polymerization reactions, enabling the production of polymers with new structures and functions. However, the one-pot synthesis of a target (macro)molecule is not simply a linear combination of each optimized reaction. The different catalytic systems used must be compatible with each other but also with the solvent, substrate, and reaction side products in order to obtain high activity and selectivity. A one-pot synthesis is thus not only a useful methodology to follow for the production of (macro)molecules, but also a promising green approach for polymer synthesis.

Poly(meth)acrylates are a major class of commodity plastics. Numerous studies have led to the discovery of multiple commercial applications for poly(meth)acrylates ranging from functional coatings to energy storage materials, high-performance engineering plastics and biomaterials. The diversity of pendant ester groups that can be inserted into the (meth)acrylic repeat unit is one of the features that allows poly(meth)acrylates to exhibit varied properties. Due to the vast number of alcohols that can act as precursors of (meth)acrylate ester monomers, the potential number of unique poly(meth)acrylates is large and only a small part of this extensive series of polymers has been investigated. This widely unexplored polymer library offers the possibility to identify original materials with interesting properties, particularly from renewable resources. Fully sustainable poly(meth)acrylates can nowadays theoretically be obtained by producing (meth)acrylic acid from renewable resources efficiently coupling it with biobased alcohols and polymerizing the resulting monomer. However, most research groups investigating the properties of biobased poly(meth)acrylates usually prepare their materials stepwise, starting from acryloyl chloride or methacrylic anhydride as these procedure require only a simple workup. Although one of the methods of choice for modifying poly(meth)acrylates properties remains copolymerization, no examples of copolymerization of (meth)acrylate derivatives from carboxylic acid precursors have yet been reported via a one-pot procedure. Herein we present a practical route to biobased poly(meth)acrylates by way of a one-pot reaction using simple commercial catalysts and we demonstrate that these requirements can be met using, inter alia, the synthesis of intermediate anhydride derivatives. This process provides direct access to (meth)acrylates and the corresponding (co)polymers in high yields.
Results and Discussion

Monomer Formation Sequence

In order to generate (meth)acyrylate monomers directly ready for copolymerization, the first objective of our one-pot approach was the synthesis of (meth)acrylic anhydrides from (meth)acrylic acids, able to act as intermediates for the synthesis of one or more esters (Scheme 1). This reaction is a known transformation that can only be achieved by dehydration of the starting compound under acidic conditions and at high temperature. To complete a one-pot procedure, it is therefore necessary to have an anhydride synthesis process that is efficient and produces anhydrides with a high yield. We have recently reported effective protocols for the preparation of cyclic anhydrides from the reaction of dicarboxylic acids in the presence of dialkyl dicarbonates under weak Lewis acid (LA) catalysis. Inspired by these previous results, it was envisaged that commercially available catalysts, such as magnesium chloride or triflate, could provide direct access to (meth)acrylic anhydrides with high selectivity and activity from the corresponding carboxylic acids.

By reacting two equivalents of (meth)acrylic acid with di-tert-butyl dicarbonate (Boc₂O) and a suitable catalyst, it is indeed possible to obtain quantitatively acrylic or methacrylic anhydrides. For instance, the mild Lewis acid MgCl₂ catalyzes selectively the formation of the anhydride within 20 minutes at 30°C (Table 1, entries 1&2). Magnesium triflate proved to be much slower for this reaction, reaching full conversion after 18 h (Table 1, entry 3). Traces of tert-butyl methacrylate were also observed. This by-product formation becomes even more pronounced when using strong Lewis acids (Table S1), as observed with La(OTf)₃ which cannot convert all the acid and only achieves 86% selectivity (Table 1, entry 4).

Acylation with anhydrides

Encouraged by these first results, we investigated the next step of our one-pot approach: the acylation of a biobased alcohol with (meth)acrylic anhydride. We hypothesized that triflate complexes would have the potential to act as catalysts given their unique robustness and versatility, as well as their activity in the acylation of alcohol. The catalytic performances of different triflate complexes were therefore evaluated in the presence of commercially available alcohols and biobased building blocks demonstrating generality of the methodology.
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this reaction, although the selectivity was lower (ca. 97%, Table S3, entries 1&2). Acrylic acid is esterified under the same conditions, although it requires a longer reaction time for La(OTf)₃ than MgCl₂, with 87 and 15 h, respectively (Table S3, entries 3&4). These two catalysts are in fact quite complementary to produce a diverse library of (meth)acrylates. On the one hand, La(OTf)₃ is indeed more selective than MgCl₂ for the esterification of MAA with bulky (and less reactive) secondary alcohols such as L-menthol (Table 3, entry 4 and Table S3, entry 5). On the other hand, MgCl₂ is more functionally tolerant, as it could selectively produce methacrylates of ethyl-L-lactate and vanillin (Table 3, entries 5&6). It should be noted that the use of a slight excess of alcohol and Boc₂O (ca. 1.2 × [Acid]) is mandatory to achieve complete conversion of (meth)acrylic acid, as the secondary reaction involving an alcohol attacking the activated Boc₂O to produce an unsymmetrical carbonate is observed to a small extent. Finally, although the use of amines in combination with Boc₂O is unsuitable (Table S3, entry 6), our methodology however makes it possible to obtain methacrylamides together with another methacrylate, by first the reaction of the amine with the anhydride and then esterification of the remaining acid.

To verify the mechanistic pathway during the first three steps, we performed the ¹H NMR kinetic monitoring of the esterification of methacrylic acid by tetrahydrogeraniol, in the presence of La(OTf)₃ or MgCl₂ (Table 3, entries 2&3). The nature of the resulting intermediates was assessed by
### Table 3: Catalytic esterification of methacrylic acid with different alcohols, dicarbonates and catalysts.\(^{[a]}\)

<table>
<thead>
<tr>
<th>Entry</th>
<th>ROC₂O</th>
<th>Alcohol</th>
<th>Catalyst (Loading)</th>
<th>(T) [°C]</th>
<th>Time [h]</th>
<th>Selectivity [%] (^{[a]})</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Moc₂O²⁻</td>
<td>–</td>
<td>Y(OTr)²⁻ (2 mol %)</td>
<td>40</td>
<td>4</td>
<td>&gt; 99</td>
</tr>
<tr>
<td>2</td>
<td>Boc₂O</td>
<td>THG</td>
<td>La(OTr)²⁻ (0.5 mol %)</td>
<td>30</td>
<td>9</td>
<td>&gt; 99</td>
</tr>
<tr>
<td>3</td>
<td>Boc₂O</td>
<td>THG</td>
<td>MgCl₂ (4 mol %)</td>
<td>30</td>
<td>15</td>
<td>98</td>
</tr>
<tr>
<td>4</td>
<td>Boc₂O</td>
<td>LMM</td>
<td>La(OTr)²⁻ (0.5 mol %)</td>
<td>30</td>
<td>41</td>
<td>96</td>
</tr>
<tr>
<td>5</td>
<td>Boc₂O</td>
<td>EL</td>
<td>MgCl₂ (4 mol %)</td>
<td>40</td>
<td>7</td>
<td>&gt; 99</td>
</tr>
<tr>
<td>6</td>
<td>Boc₂O</td>
<td>Van</td>
<td>MgCl₂ (4 mol %)</td>
<td>40</td>
<td>84</td>
<td>&gt; 99</td>
</tr>
</tbody>
</table>

(a) All reactions were performed under argon in acetonitrile. [Methacrylic Acid]=1.4 molL⁻¹ for entries 2 to 6. [ROC₂O]=[Alcohol]=1.2 x [Acid] for all entries. [b] Selectivity of the corresponding methacrylate was determined by \(^1\)H NMR spectroscopy, calculating the integral ratio of the signals of the vinylic protons of the methacrylates involved, methacrylic acid and methacrylic anhydride. [c] 1.2 equivalent of Moc₂O with respect to methacrylic acid, added sequentially after the end of the first acylation step: 0.5 equivalents at \(t=0\), 0.25 equivalents at \(t=0.5\) h, 0.25 equivalents at \(t=1.5\) h, 0.2 equivalents at \(t=3\) h.

### Table 4: Radical polymerization of various monomer mixtures with different control agents.\(^{[a]}\)

<table>
<thead>
<tr>
<th>Entry</th>
<th>M₁-M₂</th>
<th>AIBN [mol %]</th>
<th>Control Agent [mol %]</th>
<th>(X_{\text{M}1}-X_{\text{M}2}) [%] (^{[b]})</th>
<th>(M_n^{\text{exp}}) (Mₘe) [g/mol] (^{[c]})</th>
<th>(D_\text{HI})</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>LMA-</td>
<td>0.5</td>
<td>–</td>
<td>85–90</td>
<td>35 600</td>
<td>2.0</td>
</tr>
<tr>
<td>2</td>
<td>MMA</td>
<td>0.5</td>
<td>–</td>
<td>83–95</td>
<td>27 200</td>
<td>2.7</td>
</tr>
<tr>
<td>3</td>
<td>ELA-EL</td>
<td>0.5</td>
<td>–</td>
<td>97–99</td>
<td>57 500</td>
<td>1.7</td>
</tr>
<tr>
<td>4</td>
<td>LMA-</td>
<td>0.25</td>
<td>DDM (4)</td>
<td>71–75</td>
<td>5 000</td>
<td>1.5</td>
</tr>
<tr>
<td>5</td>
<td>MMA</td>
<td>0.5</td>
<td>CPDB (1.5)</td>
<td>82–88</td>
<td>9 800</td>
<td>1.1</td>
</tr>
<tr>
<td>6</td>
<td>LTHMA</td>
<td>0.5</td>
<td>CPDB (3)</td>
<td>97</td>
<td>6 200</td>
<td>1.2</td>
</tr>
<tr>
<td>7</td>
<td>LTHGA</td>
<td>0.25</td>
<td>CPDB (0.75)</td>
<td>87</td>
<td>16 300</td>
<td>1.2</td>
</tr>
</tbody>
</table>

(a) All reactions were performed under Ar, adding to the previously prepared monomer mixture in acetonitrile a solution of AIBN in toluene \((V_{\text{AIBN}}/V_{\text{Toluene}}=3)\) and a control agent, and heating at \(T=70^\circ\)C for 20 h. [b] Conversion was determined by \(^1\)H NMR spectroscopy, calculating the integral ratio of the signals of the allyl ester protons of the monomers and the polymers formed. [c] \(M_n^{\text{exp}}\) and \(D\) of polymer determined by SEC-RI in THF calibrated with polystyrene standards at 35 °C. \(M_m^{\text{exp}}\): for detailed calculations see the Supporting information.

### Polymer Formation Step

With an efficient and quantitative synthesis of (meth)acrylates in hand, we then explored the radical polymerization of the resulting monomer mixtures using 2,2′-azobis(2-methylpropionitrile) (AIBN) and various control agents. To assess the feasibility of the overall process, we conducted preliminary experiments with the rare earth triflate catalysts capable of performing the first two steps with a [catalyst]/[AIBN] ratio of 2:1 (Table 4, entry 1 and Table S4, entries 1–3). We first tested the copolymerization of LMA with MMA. Indeed, these copolymers could be of great interest to industry, since the resulting poly(meth)acrylate will have “soft” (or low \(T_g\)) segments of LMA associated with “hard” (higher \(T_g\)) segments of MMA. Remarkably, all three one-pot systems were active for the polymerization step and exhibited comparable reactivities. Also, a similar experiment using MgCl₂ for the monomer formation steps yielded comparable results (Table S4, entry 4). As a control experiment, we then performed a polymerization reaction using a clean combination of isolated methacrylates in the presence of AIBN (Table S4, entry 5). In marked contrast to what has been observed for other LA-mediated radical polymerizations of methacrylates,\(^{[21]}\) we noticed that direct polymerization gives poly(MMA-co-LMA) with a reactivity (i.e., molar masses and reaction rates) close to that obtained with one-pot systems.\(^{[22]}\) A conventional free radical pathway can also be suggested for these one-pot polymerizations, as the polymers obtained with

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Table 5: One-pot synthesis of various monomers and their block copolymerization.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Cat.</th>
<th>Control agent</th>
<th>M₁ [Mn]</th>
<th>Mₙ,exp [g/mol]</th>
<th>D</th>
<th>M₂</th>
<th>Mₙ,exp [g/mol]</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>La(OTf)₃</td>
<td>CPDB</td>
<td>L-MnMA</td>
<td>92</td>
<td>8300</td>
<td>1.3</td>
<td>THGMA</td>
<td>92</td>
</tr>
<tr>
<td>2</td>
<td>La(OTf)₃</td>
<td>CPDB</td>
<td>L-MnMA</td>
<td>97</td>
<td>6200</td>
<td>1.2</td>
<td>LMA</td>
<td>96</td>
</tr>
<tr>
<td>3</td>
<td>MgCl₂</td>
<td>CPDB</td>
<td>VMA</td>
<td>96</td>
<td>3800</td>
<td>1.1</td>
<td>ELMA</td>
<td>96</td>
</tr>
<tr>
<td>4</td>
<td>MgCl₂</td>
<td>CPDB</td>
<td>VMA</td>
<td>86</td>
<td>7400</td>
<td>1.4</td>
<td>LMA</td>
<td>75</td>
</tr>
<tr>
<td>5</td>
<td>MgCl₂</td>
<td>CPDTC</td>
<td>THGMA</td>
<td>90</td>
<td>6900</td>
<td>1.2</td>
<td>ELA</td>
<td>98</td>
</tr>
<tr>
<td>6</td>
<td>MgCl₂</td>
<td>CPDTC</td>
<td>ELMA</td>
<td>90</td>
<td>16200</td>
<td>1.6</td>
<td>THGMA</td>
<td>95</td>
</tr>
</tbody>
</table>

[a] All reactions were performed under Ar, adding to the previously prepared monomer mixture in acetonitrile a solution of AIBN in toluene (VHEMA/VAc = 3) and a control agent ([Control agent]/[AIBN] = 5), and heating at T = 70°C for 20 h. Then, the reaction mixture was cooled back to 30 or 40°C (depending on the monomer) and (meth)acrylic acid, Boc₂O and the desired alcohol were introduced (same ratios as in the 1st step). Finally, after the desired amount of time, a solution of AIBN in toluene is added and the mixture is heated at T = 70°C for 20 h. [b] Conversion was determined by ¹H NMR spectroscopy, calculating the integral ratio of the signals of the alkyl ester protons of the monomers and the polymers formed. [c] M,n,exp and D of polymer determined by SEC-RI in THF calibrated with polystyrene standards at 35°C. [d] [Control agent]/[AIBN] = 3. [e] [Control agent]/[AIBN] = 12.

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based on a degenerative transfer mechanism, this can be avoided by using a higher [RAFT agent]/[initiator] ratio, as the number of living chains is dictated by the initial number of chain transfer agent. Finally, in order to determine topology and end groups of the copolymers, a diblock copolymer poly(VMA-b-ELMA) was characterized by MALDI-ToF-MS (Figures S7–S9). Analysis of the major isotope distributions confirmed the presence of the block copolymers with cyanopropyl and thiol end-groups.[23]

New homo- and copolymers synthesized by our one-pot process were then characterized by differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA) (Table 6 & S6). The important range of $T_g$ above confirms the wide variety of characteristics that can be obtained using our synthetic method. Starting from acrylic acid, methacrylic acid or both, coupling it with various bio-based alcohols (or amines) to obtain (random or block) homo- or copolymers, the possibilities are numerous. For instance, the homopolymer of ethyl-L-lactate methacrylate displays a $T_g$ at 47°C, while the homopolymer of tetrahydrogeraniol acrylate has a glass transition at −61°C, due to its more flexible side chain and the less rigid nature of the polyacrylate backbone (Table 6, entries 1&2). The $T_g$ of poly(ELMA) could either be increased or decreased by random copolymerization of ethyl-L-lactate methacrylate with other suitable comonomers (Table 6, entries 4&5). Also, all of our di- and tri-block copolymers exhibited several glass transition temperatures, instead of a single $T_g$ for fully miscible copolymers (Table 6, entries 6 to 8). Interestingly, the significant increase in the glass transition temperature of the VMA-ELMA copolymer supports the hypothesis that the $T_g$ of these copolymers is strongly dependent on their aromatic nature (Table 6, entry 6). In addition, the double bonds in these structures can provide a functional handle for subsequent modification or cross-linking of the material. For poly(ELMA-b-THGA), two glass transitions were observed at −50°C and 40°C (Table 6, entry 7). As compared to the $T_g$s of poly(THGA) (−61°C) and poly(ELMA) (47°C) homopolymers, the small shifts indicate that the blocks of poly(THGA) and poly(ELMA) are only slightly miscible with each other. An increased thermal stability is observed for this copolymer when compared to poly(ELMA) and poly(THGA) (Figure S10). The block copolymer shows indeed a 5% weight-loss temperature of 291°C, much higher than the ones of its respective homopolymers (248°C and 272°C for poly(ELMA) and poly(THGA), respectively). Such a synergy between these two blocks is noteworthy and provides better processability to the final material, as the operational window between the second glass transition temperature and the degradation temperature is expanded. The microphase separation was also confirmed for the triblock copolymer poly(ELMA-b-THGMA-b-ELMA), since two transitions are clearly observed, which are characteristic of the glass transition of the THGMA soft phase at the lower temperature (−31°C) and the transition of the ELMA hard phase at a higher temperature (28°C) (Table 6, entry 8).

Remarkably, all homopolymers and random copolymers described in this study were colorless when using AIBN alone or AIBN and DDM as the initiating system. As expected, the RAFT agents used to synthesize the block copolymers imparted their color to the final material (yellow to pale yellow for CPDTC or pink to slightly orange for CPDB at low loadings, see Figure 2). If necessary, color removal is in principle feasible, as Perrier et al. reported an efficient method for end-group modification and chain transfer agent recovery from polymethacrylates synthesized by the RAFT process.[29]

Finally, the environmental impact of our one-pot methodology was quickly assessed by determining the E-factor of the overall synthesis and comparing it to existing literature. For instance, Epps and co-workers reported an elegant synthesis of a block copolymer of lauryl methacrylate and vanillin methacrylate, by RAFT polymerization, via a stepwise method.[30] Their work was highlighted by the promising properties displayed by these new materials, but they also rightfully noted that the E-factor of their synthesis path could be improved (estimated at 500, which did not even include monomers synthesis). We could prepare a similar polymer (Table 5, entry 4) in one-pot fashion and estimated the E-factor of the overall process, including monomers synthesis, to approximately 150 (see Supporting information for detailed calculations). This dramatic decrease in mass intensity is due to the fact that workup solvents account for the major part of the total E-factor. Avoiding monomers and homopolymers isolation is therefore key for reducing the environmental impact of a synthesis path, a feature that is inherently accomplished by one-pot methodologies.

**Conclusion**

A new one-pot synthetic route for the production of (meth)acrylate monomers and the corresponding (co)polymers has been developed from renewable feedstocks. This approach makes it possible to directly obtain biobased materials in the form of homopolymers, or random or block copolymers, without needing to isolate and purely intermediates. In addition, these catalytic systems are remarkably robust, thus allowing the use of unpurified monomers and bench-top reaction setup. In this regard, the first steps can be performed under ambient air, although maintaining an inert atmosphere is essential for the control of the subsequent

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**Table 6:** Thermal analyses of polymers obtained by one-pot catalysis,[26] Entry | Type of (co)polymer | $M_{n, exp}$ | $T_g$ | $T_g$ | $T_{5\%}$ |
--- | --- | --- | --- | --- | --- |
1 | poly(ELMA) | 43 900 | 47 | 247 | 247 |
2 | poly(THGA) | 25 000 | −61 | 272 | 272 |
3 | poly(THGMA) | 38 300 | −27 | 214 | 214 |
4 | poly(ELMA-co-MMA) | 37 000 | 74 | 247 | 247 |
5 | poly(ELMA-co-ELA) | 60 300 | 27 | 310 | 310 |
6 | poly(THGMA-co-ELMA) | 28 100 | 11 | 40 | 226 |
7 | poly(ELMA-co-THGMA) | 24 100 | 40 | −50 | 291 |
8 | poly(THGMA-co-THGMA-co-ELMA) | 22 100 | 28 | −31 | 208 |

[a] $M_{n, exp}$ of polymer determined by SEC-RI in THF calibrated with polystyrene standards at 35°C. $T_g$ of polymer determined by DSC on second heating cycle (10°C/min, N2 flow). $T_{5\%}$ of polymer determined by TGA (20°C/min, N2 flow).
polymerization step. Ultimately, the strategy provides easy access to a set of unique macromolecular structures that can be used to meet the growing demand for new applications for commercial polymers. Our future efforts are oriented towards further study of the reaction mechanism, as well as development of catalysts that exhibit higher reactivities for the whole process.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: (meth)acrylates · biobased polymers · one-pot catalysis · reaction mechanisms · renewable monomers


[23] In a typical free radical polymerization, the stereoregulating effect results from a steric interaction between the incoming alkene and nearest stereocenter, but due to the conformational mobility of the chain, this effect is minimal: For example, the ratio of kᵣ/kₛ, for methyl acrylate is 1:1 at 0°C: a) P. Pino, U. W. Suter, Polymer 1976, 17, 977–995; b) T. Ando, M. Kamigaito, M. Sawamoto, Macromolecules 1997, 30, 4507–4510.


[27] G. Gody, T. Mashmeyer, P. B. Zetterlund, S. Perrier, Nat. Commun. 2013, 4, 2505; [■ article number ok?][■]


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Research Articles

Biobased Polymers

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We have developed a multicatalytic approach for the one-pot production of (meth)acrylate monomers, allowing the formation of the corresponding (co)polymers from renewable feedstocks. This highly efficient strategy allowed us to perform multiple catalytic transformations in one pot, while bypassing several purification steps. This procedure can therefore minimize chemical waste, save time, and simplify practicalities.

Biobased Polymers

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