

## SYNTHESIS OF POLY(GLYCIDYL METHACRYLATE) VIA MECHANO-ATRP USING BARIUM TITANATE AND ZINC OXIDE NANOPARTICLES AS ACTIVE AGENTS

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### Abstract

This study focuses on the synthesis of poly(glycidyl methacrylate) (PGMA) using a mechanically controlled atom transfer radical polymerization (mechano-ATRP) activated by barium titanate (BaTiO<sub>3</sub>) or zinc oxide (ZnO) nanoparticles as active agents. Polymerization optimizations were performed with a typical ATRP reaction mixture containing monomer, initiator, ligand, and transition metal. The piezoelectric agents were used at the amounts of 0.5 wt.% for ZnO and 4.5 wt.% for BaTiO<sub>3</sub> nanoparticles. The presence of oxygen in the reaction mixture was minimized through argon purging. The mechano-ATRP was carried out in an ultrasonic bath under defined conditions, such as intensity, for 5 hours. The reaction kinetics were evaluated using proton nuclear magnetic resonance (<sup>1</sup>H NMR) by calculating monomer conversion over time. The polydispersity index and molar mass of the polymer were determined using gel permeation chromatography (GPC). The aim of this study was to gain control over the mechano-ATRP of GMA to produce a well-defined polymer with a low polydispersity index, specifically below 1.4, and sufficient conversion of around 40%.

**Keywords:** Polymer chemistry, control radical polymerization, glycidyl methacrylate, mechanochemistry, mechano-ATRP

### 1. INTRODUCTION

Controlled radical polymerization techniques have emerged as indispensable tools for the synthesis of polymers with well-defined architectures [1-3], narrow molecular weight distributions [4-6], and tailored functionalities [7-8]. Among them, atom transfer radical polymerization (ATRP) has attracted particular attention due to its robustness and versatility in mediating the polymerization of a wide range of monomers [9]. Traditionally, ATRP relies on transition metal complexes under thermal [10] or photochemical activation [11]; however, the need for more sustainable, energy-efficient, and externally controllable methods has stimulated the development of alternative initiation strategies.

In recent years, mechanochemical and mechano-chemical-electrical coupling approaches have provided a promising route for conducting ATRP under mild conditions [12]. In particular, mechano-ATRP harnesses mechanical forces to generate redox-active species capable of maintaining the ATRP equilibrium, enabling polymerizations without the need for continuous heating or high-energy radiation [13]. The concept was first reported in 2016 by Esser-Kahn and co-workers, who demonstrated that piezoelectric nanoparticles could mediate ATRP under ultrasonic agitation by converting mechanical energy into catalytic redox activity [14].

This seminal work established the foundation for a new class of externally regulated ATRP processes and inspired further exploration into piezoelectrically driven polymerizations [15].

Piezoelectric oxides, such as barium titanate ( $\text{BaTiO}_3$ ) and zinc oxide ( $\text{ZnO}$ ), are particularly attractive in this context due to their high piezoelectric coefficients, chemical stability, and low cost. Both materials have been reported as efficient piezoactive agents for driving redox transformations under ultrasonic or mechanical agitation [16-18].

The polymerization techniques are currently focused on the typical monomers such as methyl acrylate, ethyl acrylate, tert-butyl acrylate, n-butyl acrylate, and methyl methacrylate [19]. Due to the abovementioned research, there is a scientific gap in glycidyl methacrylate (GMA) polymerized using the mechano-ATRP approach. This monomer, bearing a reactive epoxide side chain, offers a valuable platform for post-polymerization modifications [20], enabling the design of advanced functional materials [21], coatings [22], and biomedical systems [23]. Achieving controlled polymerization of GMA via mechano-ATRP would therefore represent a significant step forward in the development of piezoelectrically mediated polymerization strategies.

Herein, we report the mechano-ATRP of GMA using  $\text{BaTiO}_3$  and  $\text{ZnO}$  nanoparticles as piezoactive agents. We demonstrate that the piezoelectric response of these oxides under ultrasonic excitation is capable of mediating the ATRP process, yielding poly(glycidyl methacrylate) with predictable molecular weights and controlled dispersities. The effects of the piezoactive material type, loading, and reaction conditions are systematically investigated, providing insight into the roles of  $\text{BaTiO}_3$  and  $\text{ZnO}$  in facilitating redox control. This study highlights the potential of piezoelectric nanomaterials in enabling sustainable controlled polymerization and expands the toolbox of mechano-ATRP toward functional methacrylate systems.

## 2. MATERIALS AND CHARACTERIZATIONS

The monomer GMA (glycidyl methacrylate), initiator EBiB (ethyl  $\alpha$ -bromo-isobutyrate), ligand TPMA (tris(2-pyridylmethyl)amine), and transition metal  $\text{CuBr}_2$  (copper(II) bromide) were purchased from Sigma-Aldrich. As for piezoelectric carriers,  $\text{ZnO}$  was synthesized using microwave (MW)-assisted polyol-mediated synthesis.  $\text{BaTiO}_3$  particles  $< 3 \mu\text{m}$  were purchased from Sigma-Aldrich as well. The monomer was purified by passing through a column of basic alumina, cotton, and a  $0.22 \mu\text{m}$  PTFE syringe filter to remove inhibitors. All other chemicals were used as received.

1.6 mL of GMA (11.7 mmol, 100 equiv.), 17.2  $\mu\text{L}$  of EBiB (1.17 mmol, 1 equiv.), 5.4 mg of TPMA (0.19 mmol, 0.16 equiv.), 1.05 mg of  $\text{CuBr}_2$  (0.05 mmol, 0.04 equiv.), and 2.223 mL of DMSO (58vol%) were added into a 4.8 mL vial, and piezoactive particles were added. The particle loading was calculated with respect to monomer and solvent mass, and was added to the reaction vial (185.1 mg, 4.5 wt.% for  $\text{BaTiO}_3$  and 20.5 mg, 0.5 wt.% for  $\text{ZnO}$ , respectively). To assess the effects of oxygen, mixtures were prepared under two conditions: argon-purged or sealed in reactors with residual oxygen. The reaction was then exposed to ultrasound agitation in Bandelin Sonorex RK 106 with a frequency of 35 kHz and Elma Transsonic digital T490 DH with a frequency of 40 kHz. The temperature was set to  $30^\circ\text{C}$  and was maintained stable using a thermostat Julabo F12-ED.

Samples were withdrawn using syringes at intervals of 1, 3, and 5 hours. These samples were then diluted using d-chloroform for proton nuclear magnetic resonance ( $^1\text{H}$  NMR) spectroscopy to determine conversion of monomer in time (kinetics) and tetrahydrofuran (THF) for gel permeation chromatography (GPC) to determine average molecular weight ( $M_n$ ) and dispersity ( $M_w/M_n$ ), respectively.

$^1\text{H}$  NMR was performed on a JEOL ECZ400R/S3 spectrometer (JEOL, USA) and GPC on a Waters HPLC e2695 with RI detector 2414 and equipped with: PL gel MIXED-A ( $300 \times 7.5 \text{ mm}$ ,  $20 \mu\text{m}$ ), PL gel MIXED-B ( $300 \times 7.5 \text{ mm}$ ,  $10 \mu\text{m}$ ), and PL gel MIXED-D ( $300 \times 7.5 \text{ mm}$ ,  $5 \mu\text{m}$ ). The flow rate was set to  $1 \text{ mL} \cdot \text{min}^{-1}$  at  $40^\circ\text{C}$ . Apparent molecular weights were determined using polystyrene (PS) standards.

### 3. RESULTS AND DISCUSSION

In total, 8 reactions were performed using two different active agents and two different ultrasonic baths. ZnO and BaTiO<sub>3</sub> were selected based on the previous works of Matyjaszewski [19] and Cvek [15]. Oxygen removal was alternated between reactions to determine the influence of oxygen on the reaction control. Polymerization of GMA in the presence of 0.5 wt% of ZnO using a 35 kHz ultrasonic bath yielded a polymer with 36-38% conversion and reasonable dispersity of 1.24-1.26 (**Table 1**, Entries 1-2). The plot of  $M_{n\text{GPC}}$  vs. conversion showed a linear growth of polymer molar mass, although the trend was shifted upward. The values of  $M_{n\text{GPC}}$  consistently were higher than the  $M_n$  theoretical, which could be attributed to slow initiation or termination via exchange processes (**Figure 1a**).

**Table 1** Results for mechano-ATRP of GMA mediated with ZnO or BaTiO<sub>3</sub> as piezoactive catalysts

Entry <sup>a</sup>	PA	Loading (wt.%)	f (kHz)	$M_n$ GPC <sup>b</sup> (g·mol <sup>-1</sup> )	$M_n$ theor. <sup>c</sup> (g·mol <sup>-1</sup> )	$\mathcal{D}$ <sup>b</sup> (-)	Conversion <sup>d</sup> (%)
1	ZnO	0.5	35	11000	5100	1.26	36
2 <sup>e</sup>	ZnO	0.5	35	11200	5300	1.24	30
3	ZnO	0.5	40	9800	4400	1.22	31
4 <sup>e</sup>	ZnO	0.5	40	11400	2700	1.26	19
5	BaTiO <sub>3</sub>	4.5	35	8900	6500	1.22	45
6 <sup>e</sup>	BaTiO <sub>3</sub>	4.5	35	7500	4200	1.25	30
7	BaTiO <sub>3</sub>	4.5	40	6200	6200	1.22	43
8 <sup>e</sup>	BaTiO <sub>3</sub>	4.5	40	6300	3700	1.20	26

<sup>a</sup>Reaction conditions: [GMA]<sub>0</sub>/[EBiB]<sub>0</sub>/[TPMA]<sub>0</sub>/[CuBr<sub>2</sub>]<sub>0</sub> = 100:1:0.16:0.04, 58vol% DMSO, temperature 30°C

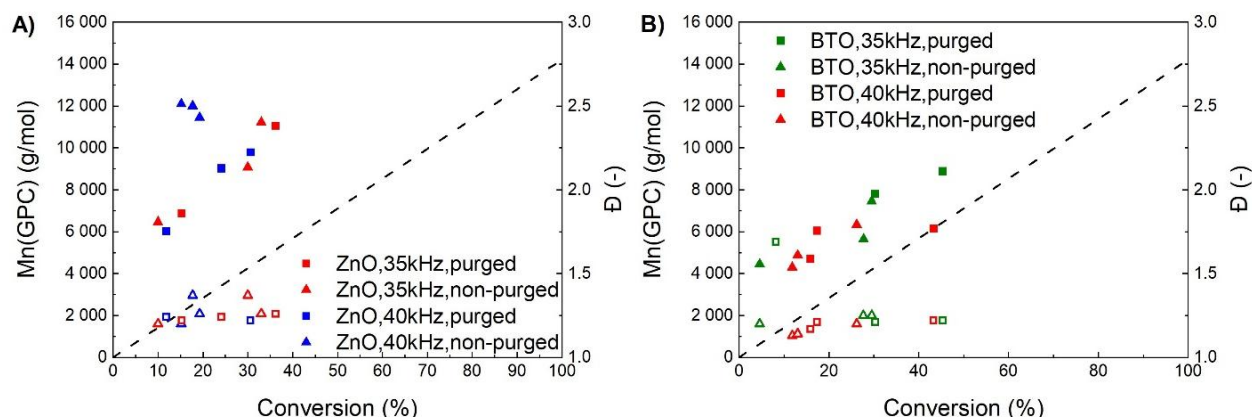
<sup>b</sup>Molar mass  $M_n$  and dispersity  $\mathcal{D}$  were determined by GPC using THF as eluent. Calibration was done with PS

<sup>c</sup>Theoretical value of molar mass was calculated using the equation  $M_n \text{ theor.} = c \cdot M_{\text{WGMA}} + M_{\text{WEBiB}}$ , where  $c$  represents conversion of monomer,  $M_{\text{WGMA}}$  molar mass of monomer,  $M_{\text{WEBiB}}$  molar mass of initiator

<sup>d</sup>Conversion of monomer was calculated based on <sup>1</sup>H NMR analysis; <sup>e</sup>the reaction was not purged with argon.

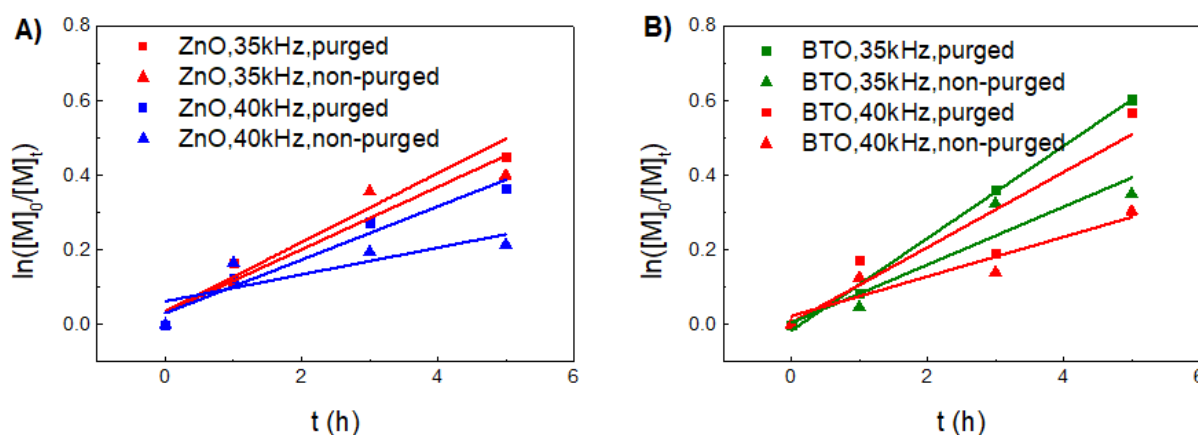
The other reaction in the presence of 0.5 wt.% ZnO using a 40 kHz ultrasonic bath, interestingly, demonstrated a pronounced impact of oxygen contamination. From 31% conversion after 5 hours, it dropped to only 19 % (**Table 1**, Entries 3-4), although  $M_{n\text{GPC}}$  shows different values of 9800 g·mol<sup>-1</sup> vs. 11400 g·mol<sup>-1</sup>, respectively. Values obtained from GPC were once again higher than the  $M_n$  theoretical (**Figure 1a**). In the non-purged reaction,  $M_{n\text{GPC}}$  values increased rapidly to approx. 12000 g·mol<sup>-1</sup> within the first hour and then slightly decreased to 11400 g·mol<sup>-1</sup>. This behavior suggests that the presence of dissolved oxygen led to rapid radical deactivation and premature termination of chain growth.

Reactions conducted in the presence of BaTiO<sub>3</sub> showed higher overall conversions (up to 45% after 5 hours; **Table 1**, Entry 5) and lower dispersion ( $\mathcal{D}$  value of 1.20 after 5 hours; **Table 1**, Entry 8). The influence of oxygen is apparent in both cases, lowering conversions from 45% to 30% with a 35 kHz ultrasonic bath and 43% to 26% with a 40 kHz ultrasonic bath, respectively (**Table 1**). Values  $M_{n\text{GPC}}$  were closer to  $M_n$  theoretical and followed a linear trend for the 35 kHz bath (**Figure 1b**), but were non-linear for the 40 kHz ultrasonic bath. The value of  $M_{n\text{GPC}}$  after 5 hours is identical to the  $M_n$  theoretical, which, however, results from the fact that the  $M_{n\text{GPC}}$  did not increase between 3 to 5 hours of reaction, even though the conversion rose from 19 to 43%. Despite the application of BaTiO<sub>3</sub> improving conversions and dispersity, the reactions still exhibited clear signs of oxygen inhibition, particularly under 40 kHz ultrasound.



**Figure 1** Results from mechano-ATRP of GMA. Reaction conditions:  $[GMA]_0/[EBiB]_0/[TPMA]_0/[CuBr_2]_0 = 100:1:0.16:0.04$ , 58 vol% DMSO, temperature 30°C, A) with 0.5 wt.% ZnO particles, B) with 4.5 wt.% BaTiO<sub>3</sub> particles, solid symbols represent  $M_{n(GPC)}$  values, open symbols represent  $\bar{D}$  values

The reaction kinetics were evaluated using a semilogarithmic kinetic plot (**Figure 2**). The most controlled reaction terms of the kinetics rate constant was observed with 4.5 wt.% BaTiO<sub>3</sub> in the 35 kHz ultrasonic bath, yielding  $k[M] = 0.121 \text{ h}^{-1}$  and an  $R^2$  value of 0.9965. When performing this reaction without purging, the rate constant decreased to  $k[M] = 0.070 \text{ h}^{-1}$  (**Figure 2b**). The reaction performed with 0.5 wt.% ZnO using the 35 kHz ultrasonic bath reached a rate constant of  $k[M] = 0.090 \text{ h}^{-1}$  with an  $R^2$  value of 0.9669. The difference in catalytic efficiency between ZnO and BaTiO<sub>3</sub> can be attributed to their intrinsic properties. As is known, BaTiO<sub>3</sub> possesses a significantly higher piezoelectric coefficient than ZnO, enabling the generation of stronger local electric fields under ultrasonic stimulation. However, despite seemingly slower kinetics, the results indicate that ZnO is in fact a more reactive mediator in mechano-ATRP, as its higher activity leads to premature radical termination (even at much lower loading). These findings highlight the need for further optimization to establish conditions that enable even higher control over the mechano-ATRP of GMA.



**Figure 2** Semilogarithmic kinetic plot for mechano-ATRP of GMA A) in the presence of 0.5 wt.% ZnO and B) in the presence of 4.5 wt.% BaTiO<sub>3</sub>

#### 4. CONCLUSION

The present study demonstrates that poly(glycidyl methacrylate) can be synthesized via mechano-ATRP using selected nanoparticles as activators, with both ZnO and BaTiO<sub>3</sub> proving effective catalytic capabilities under ultrasonic stimulation. Polymerizations mediated by ZnO (0.5 wt.%) yielded good monomer conversions of approximately 36-38% and dispersity values around 1.25, whereas BaTiO<sub>3</sub> (4.5 wt.%) enabled higher

conversions of up to 45% and narrower dispersities of approximately 1.20, indicating improved control over the polymerization process in comparison to ZnO. The influence of oxygen was consistently observed, particularly in the BaTiO<sub>3</sub>-mediated systems, where its presence resulted in a more significant reduction of conversion. Kinetic analysis revealed that the most controlled reaction was obtained with BaTiO<sub>3</sub> under 35 kHz ultrasonic irradiation, which exhibited an apparent first-order rate constant of 0.121 h<sup>-1</sup> and excellent linearity ( $R^2 = 0.9965$ ), while the absence of argon purging led to a marked decrease in the rate constant. Furthermore, the correlation between number-average molecular weight and conversion was found to be linear and in close agreement with theoretical predictions under optimized conditions. Overall, these findings showed promising results and the viability of the mechano-ATRP approach as a strategy for the controlled synthesis of well-defined PGMA and underscore the crucial roles of active agent selection, ultrasonic intensity, and oxygen exclusion in achieving high levels of control.

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