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2-Adamantil Tosilat Başlatıcısı Kullanılarak 2-Etil-2-Oksazolin Monomerinin Katyonik Halka Açılma Polimerizasyonu

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ÖZET

Katyonik halka açılma polimerizasyonu (CROP) ile sentezlenen Poli(2-alkil/aril-2-oksazolin) (POx) polimerleri, biyouyumlulukları, gizlenme (stealth) özellikleri ve ayarlanabilir yapısal karakteristikleri nedeniyle önemli derecede ilgi çekmektedir. Bu çalışmada, 2-adamantil tosilat (AdTos) başlatıcısı kullanılarak poli(2-etil-2-oksazolin) (PEtOx) polimerizasyonunun kinetik özellikleri araştırılmıştır. Polimerizasyon mekanizması, monomer dönüşümü, molekül ağırlığı değişimi ve dispersite analizleri ile incelenmiş ve polimerizasyonun psödo-birinci dereceden kinetik ile gerçekleşen, iyi kontrollü bir süreç olduğu ortaya konulmuştur. Deneysel sonuçlar, teorik ve deneysel molekül ağırlıkları arasında güçlü bir korelasyon olduğunu göstermiş; zincir transferi ve sonlanma reaksiyonlarının minimum düzeyde gerçekleştiği, yaşayan bir polimerizasyon mekanizmasını doğrulamıştır. Ayrıca jel geçirgenlik kromatografisi (GPC) ve matriks destekli lazer desorpsiyon/iyonizasyon uçuş zamanlı kütle spektrometresi (MALDI-ToF MS) analizleri ile polimer zincirlerinin homojen bir büyüme gösterdiği ve uç grup bütünlüğünün korunduğu kanıtlanmıştır. Bu bulgular, katyonik halka açılma polimerizasyonuna yönelik genel bilgi birikimine katkı sağlamakta ve AdTos'un kontrollü moleküler mimariye sahip, iyi tanımlanmış PEtOx polimerlerinin sentezlenmesinde etkili bir başlatıcı olduğunu ortaya koymaktadır.

Cationic Ring Opening Polymerization of 2-Ethyl-2-Oxazoline Monomer Using 2-Adamantyl Tosylate Initiator

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ABSTRACT

Poly(2-alkyl/aryl-2-oxazoline)s (POxs) synthesized via cationic ring-opening polymerization (CROP) have gained significant attention due to their biocompatibility, stealth properties, and tunable characteristics. This study investigates the kinetic aspects of poly(2-ethyl-2-oxazoline) (PEtOx) polymerization using 2-adamantyl tosylate (AdTos) as an initiator. The polymerization mechanism was examined through monomer conversion, molecular weight evolution, and dispersity analysis, revealing a well-controlled process following pseudo-first-order kinetics. The experimental results demonstrated a strong correlation between theoretical and measured molecular weights, confirming a living polymerization mechanism with minimal chain transfer and termination reactions. Additionally, gel permeation chromatography (GPC) and matrix-assisted laser desorption/ionization time-of-flight (MALDI-ToF) mass spectrometry validated the uniform growth of polymer chains with well-defined end-group fidelity. These findings contribute to the broader understanding of CROP and highlight AdTos as an effective initiator for synthesizing well-defined PEtOx with controlled molecular architectures.

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INTRODUCTION

Cationic ring-opening polymerization (CROP) was first introduced in the 1960s for the synthesis of polyethers and polyamide-type polymers and has since become a key method for the polymerization of 2-substituted-2-oxazoline monomers [1,2]. The method attracted increasing attention due to its ability to proceed under living or quasi-living conditions, enabling precise control over the molar mass, dispersity, and architecture of the resulting polymers by minimizing undesirable side reactions such as chain transfer and premature termination [3]. CROP operates via a well-established three-step mechanism: initiation by an electrophilic species that activates the monomer, propagation through nucleophilic substitution reactions extending the polymer chain, and termination by external nucleophiles that cap the growing chain [1,2]. Under optimized conditions, the living nature of the polymerization allows the synthesis of polymers with narrow molar mass distributions and well-defined end-group functionalities. Furthermore, the versatility of the process enables fine-tuning of the polymer properties through the careful design of monomer side chains and the judicious selection of initiators and terminating agents [4].

Poly(2-alkyl/aryl-2-oxazoline)s (POxs), synthesized via the CROP of 2-substituted-2-oxazoline monomers, have emerged as a highly versatile class of polymers with rapidly growing relevance in polymer and materials science [1,2,5–8]. Their pseudopeptidic backbone structure, which closely resembles that of poly(amino acid)s, grants POxs unique physicochemical characteristics and makes them highly attractive for biomedical and pharmaceutical applications [9–11]. Notably, POxs demonstrate excellent biocompatibility [12,13], stealth-like behavior that minimizes recognition by the immune system [14,15], and remarkable structural diversity through modification of their side chains [4,16]. Furthermore, their thermal and crystalline properties are tunable over a wide range, which allows for the design of materials with specific melting points, solubilities, and mechanical profiles [17]. These features facilitate the formation of well-defined self-assembled nanostructures such as micelles, vesicles, and nanogels, which are of significant interest in drug delivery and nanomedicine [18,19]. Beyond their structural tunability, POxs also offer excellent solubility in both aqueous and organic solvents, and their solution behavior can be finely adjusted via side-chain engineering, hydrophobic/hydrophilic balance, and copolymer composition [3,20–26]. This level of control has positioned POxs at the forefront of advanced polymeric systems for targeted therapies, diagnostics, and responsive materials.

Among the various POx derivatives, poly(2-ethyl-2-oxazoline) (PEtOx) stands out as the most extensively studied due to its favorable pharmacological profile and synthetic accessibility. PEtOx has been widely investigated as a potential substitute for poly(ethylene glycol) (PEG), particularly in the design of drug delivery systems, due to its low immunogenicity, prolonged blood circulation time, and superior tumor accumulation via the enhanced permeability and retention (EPR) effect [11,13,14]. The versatility of PEtOx enables the development of a wide array of functional architectures—such as micelles, hydrogels, and polymer-drug conjugates—capable of encapsulating or covalently binding therapeutic agents [11,15,27]. These systems allow for controlled and stimuli-responsive drug release, thereby improving therapeutic efficacy while minimizing side effects. Moreover, the chemical structure of PEtOx is amenable to functionalization with various reactive groups, facilitating the design of smart delivery platforms that respond to environmental triggers such as pH, temperature, redox gradients, or enzymatic activity [13,18,20–22]. Recent studies have expanded the biomedical applications of PEtOx-based materials beyond drug delivery to include gene transfection agents, protein stabilizers, stealth coatings for nanoparticles, and components of diagnostic devices [11,13,15]. Collectively, the tunable nature, favorable biological profile, and synthetic flexibility of POxs—particularly PEtOx—underscore their immense promise as key materials in the development of next-generation biomaterials and nanotechnological systems.

A wide variety of initiator systems have been investigated for the synthesis of POxs, with significant influence on polymerization kinetics, molar mass control, and polymer end-group functionality. Among these, alkylsulfonates such as methyl p-toluenesulfonate and trifluoromethanesulfonate (triflate) have been extensively employed due to their high reactivity and ability to initiate polymerization efficiently [26,27]. p-Nitrobenzenesulfonates (nosylates) have also been utilized, offering enhanced initiation efficiency compared to traditional tosylates [26]. In addition to sulfonate esters, alkyl, benzyl, and acetyl halides have been explored, although their lower electrophilicity often results in slower polymerization rates or broader molar mass distributions [12]. Oxazolinium salts, generated in situ or pre-formed, have been shown to serve as highly reactive initiating species, enabling more controlled polymerizations under appropriate conditions [6]. Furthermore, Lewis acids such as metallocenes have been investigated as alternative initiators, providing unique control over the polymerization process, albeit requiring stricter reaction conditions [7]. While these initiator systems have greatly expanded the scope and tunability of CROP, the design of functional initiators remains challenging due to the requirement that they must be non-nucleophilic to prevent side reactions that would interfere with the living nature of the polymerization [8]. Despite extensive research into various initiators, to the best of our knowledge, the use of 2-adamantyl tosylate (AdTos) as an initiator for the polymerization of 2-oxazoline monomers has not been previously reported.

In this study, a detailed kinetic investigation was conducted to evaluate the effect of AdTos on the polymerization of poly(2-ethyl-2-oxazoline) (PEtOx). Understanding polymerization kinetics is essential for predicting polymer growth and designing controlled polymerization systems tailored for specific applications. By analyzing monomer conversion, molecular weight evolution, and dispersity trends, we provide insights into the polymerization mechanism and establish a framework for the rational design of well-defined POxs. Our findings contribute to the broader understanding of CROP and support the development of tailored polymer architectures for advanced material applications.

MATERIALS AND METHODS

Materials

2-Ethyl-2-oxazoline (EtOx, 99+%, Acros Organics) was dried over calcium hydride and distilled under reduced pressure before use. Extra dry acetonitrile (99+%, Acros Organics) was obtained and stored under an inert atmosphere. p-Toluenesulfonyl chloride (TsCl) was recrystallized from petroleum ether prior to use. All other chemicals were purchased from Sigma-Aldrich and used without further purification.

Instrumentation

^{1}H Nuclear Magnetic Resonance (NMR): All spectra were recorded on a Bruker Advance III HD 300 MHz. $CDCl_3$ was used as solvent and the signal of the residual $CHCl_3$ served as reference for the chemical shift δ expressed in ppm. Data analysis was performed using TopSpin 3.2 software.

Gel Permeation Chromatography (GPC): The measurements were performed using THF (2% TEA and 0.01% BHT) as the eluent. The analysis was conducted using an Agilent Technologies 1260 Infinity system equipped with a refractive index (RI) detector, a PLgel 5 µm guard column, and a PLgel 5 µm mixed D column (dimensions: 300 × 7.5 mm). Samples were analyzed at a flow rate of 1 mL min^{-1} and a column temperature of 40 °C. Calibration was performed using poly(methyl methacrylate) standards (Agilent PMMA calibration kits, M-M-10 and M-L-10), covering a molecular weight range of 500–120,000 Da. Before injection (100 µL), each sample was filtered through a 0.2 µm PTFE membrane. Molecular weights were calculated by conventional calibration using Agilent GPC/SEC

software, and the results were plotted with OriginPro 2022b.

Matrix-assisted laser desorption ionization–time of flight (MALDI-ToF) analysis was conducted using a Bruker Autoflex Speed mass spectrometer, equipped with a nitrogen laser emitting 2 ns pulses at a wavelength of 337 nm. Measurements were carried out in positive ion reflective mode with an accelerating voltage set to 25 kV. The matrix utilized was trans-2-[3-(4-tert-butylphenyl)-2-methyl-2-propylidene]malononitrile (DCTB), dissolved in tetrahydrofuran (THF). Potassium trifluoroacetate, dissolved in ethanol, served as the S2 cationization agent. Calibration was performed using poly(methyl methacrylate) (PMMA) standards.

Synthesis of 2-Adamantyl Tosylate Initiator

The synthesis of the 2-adamantyl tosylate (AdTos) initiator is summarized in Figure 1. A solution of 1-adamantanol (5 g, 30 mmol) in 30 mL of THF was cooled to 0°C, followed by the addition of triethylamine (TEA, 3.34 g, 33 mmol). Subsequently, p-toluenesulfonyl chloride (6.30 g, 33 mmol) was introduced, and the reaction mixture was stirred overnight at room temperature. The resulting precipitate was filtered off, and the solvent was evaporated under vacuum. The crude residue was then dissolved in dichloromethane (DCM), washed with water, dried over sodium sulfate, and concentrated by evaporating the excess solvent under reduced pressure. Finally, the crude product was purified by vacuum distillation. (7.05 g, yield 70%).

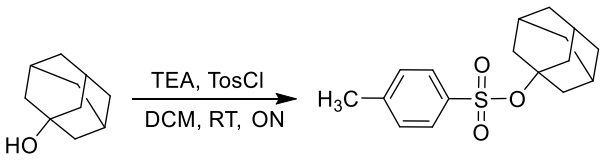

Figure 1
Reaction scheme for the synthesis of AdTos initiator.

Synthesis of PEtOx

Polymerizations were initiated using 2-adamantyl tosylate as the initiator, with a monomer-to-initiator molar ratio of 50:1. A stock solution was prepared by dissolving 7.856 g of 2-ethyl-2-oxazoline (4 M), 0.485 g of initiator, and 20.2 mL of solvent. This solution was then aliquoted into six Biotage 2–5 mL microwave vials to enable parallel experiments. The vials were sealed with a PTFE-lined butyl rubber septum and an aluminum crimp cap. A sample was taken at t0 before the vials were placed in a preheated oil bath at 120 °C for varying time intervals. The polymerization progress was monitored using ^{1}H-NMR and GPC analyses. The final polymer was purified by precipitating twice in diethyl ether.

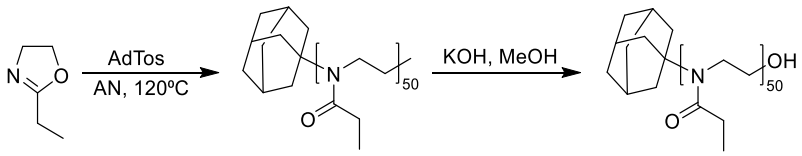

Figure 2
Reaction scheme for the AdTos initiated EtOx polymerization.

RESULTS AND DISCUSSION

This section summarizes the key findings of the polymerization study. The successful synthesis of the 2-adamantyl tosylate (AdTos) initiator is confirmed by ^{1}H NMR, followed by kinetic analysis of 2-ethyl-2-oxazoline (EtOx) polymerization, covering monomer conversion, molecular weight evolution, and dispersity. GPC and MALDI-ToF data further validate the controlled/living character of the process

and the formation of well-defined PEtOx.

^{1}H NMR Analysis of the AdTos Initiator

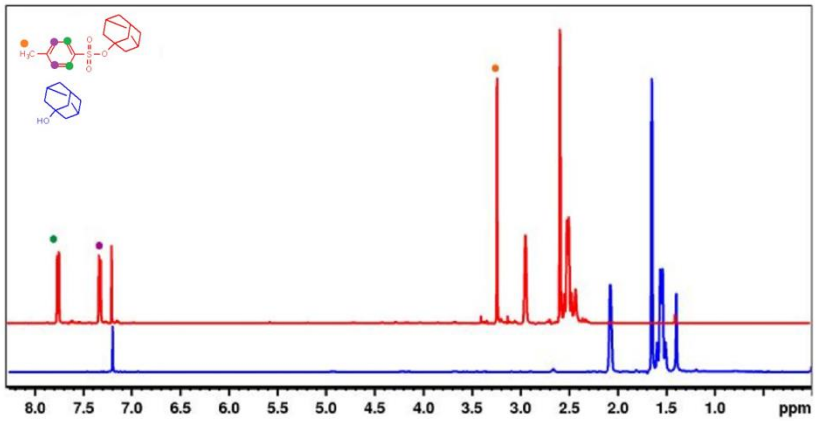

Figure 3
^{1}H NMR spectra of the 1-adamentanol and AdTos initiator.

Figure 3 presents the ^{1}H-NMR spectra of 2-adamantyl tosylate (AdTos) (red) and its precursor, 1-adamantanol (blue), recorded in $CDCl_3$ (deuterated chloroform). The aromatic proton signals between 7.3–7.8 ppm in the AdTos spectrum is characteristic of the p-toluenesulfonyl (TsO) functional group, which is absent in the 1-adamantanol precursor. The adamantane proton signals (~1.5–2.2 ppm) remain unchanged, confirming that the adamantyl core structure is preserved throughout the tosylation reaction. Additionally, the appearance of the methyl ($-CH_3$) signal of the tosyl group around 2.4–2.5 ppm further supports the successful conversion of 1-adamantanol to AdTos.

Kinetic of Polymerization

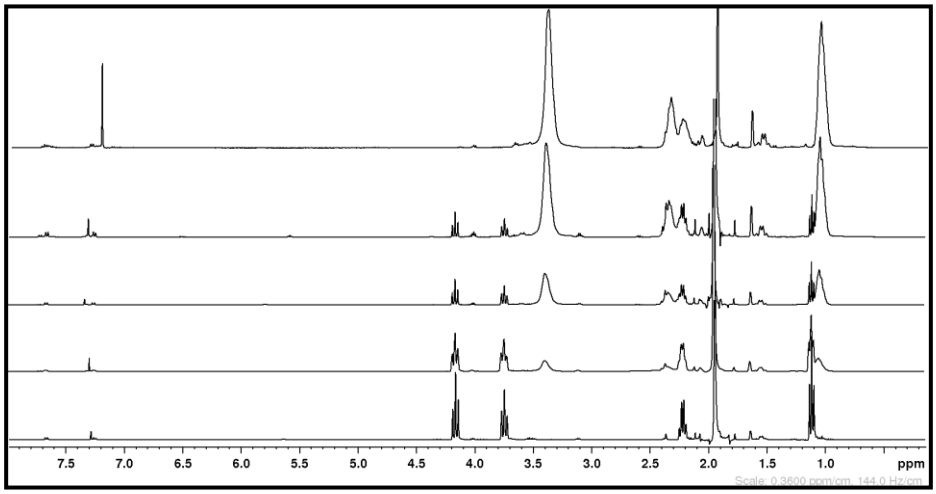

Figure 4
^{1}H NMR data for conversion of 2-adamantyl tosylate initiated polymerization of 2-ethyl oxazoline (EtOx) at 120°C.

The ^{1}H-NMR spectrum confirms the successful polymerization of 2-ethyl-2-oxazoline (EtOx) using 2-adamantyl tosylate (AdTos) as the initiator (Figure 4). The characteristic backbone peaks observed in the 3.3–4.0 ppm range correspond to the methylene ($-CH_2-$) protons in the polymer chain, while the signals in the 1.0–1.5 ppm region confirm the presence of ethyl side chains. Notably, the 3.3–4.0 ppm backbone peak increases in intensity as the reaction progresses, while the monomer peaks at 4.1–4.3 ppm diminish correspondingly. This trend indicates the successful conversion of

monomers into polymer, demonstrating that the polymerization proceeds efficiently over time. The adamantyl end-group is evident from peaks appearing between 1.6–2.2 ppm, suggesting effective initiation. Minor signals in the 7.0–7.5 ppm range correspond to the p-toluenesulfonate (TsO^-) group, and the conversion can be calculated based on the monomer and TsO^- peak intensities for further calculations of theoretical Mn. The near-complete disappearance of monomer-related peaks further supports the high conversion of EtOx.

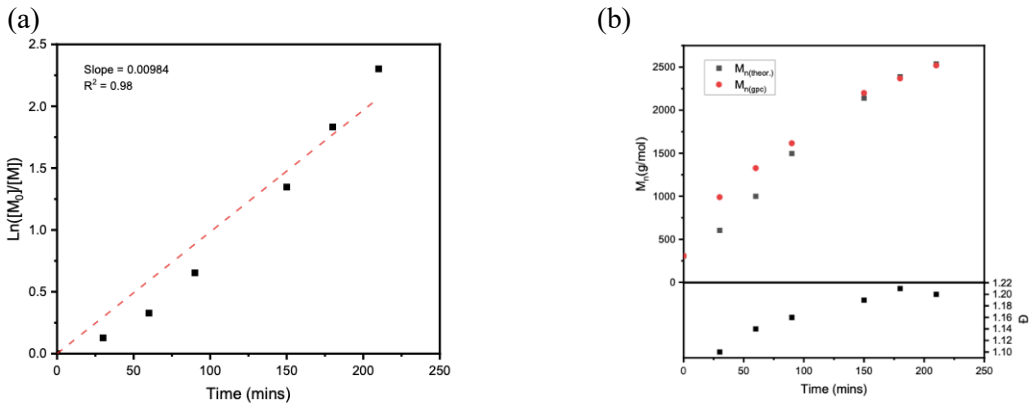

Figure 5

Kinetics for the polymerization of EtOx (a), evolution of molecular weight and dispersity over conversion for CROP of PEtOx (b). All the polymerizations were performed at 120 °C in MeCN with a 4 M total monomer concentration with 2-adamantyl tosylate as an initiator.

Figure 5a presents the kinetic plot for the polymerization of EtOx initiated by AdTos. The natural logarithm of the monomer concentration ratio (ln([M0]/[M]) is plotted as a function of time, exhibiting a linear trend with a high correlation coefficient (R^2=0.98R). This strong linearity confirms that the polymerization follows pseudo-first-order kinetics with respect to monomer consumption.

The slope of the linear fit yields a polymerization rate constant of k_p=0.00984 min^{-1} under the given reaction conditions. This result suggests a well-controlled polymerization process, where the monomer is consumed at a predictable rate, allowing for precise control over molecular weight. The high degree of linearity further supports the absence of significant termination or chain transfer reactions during polymerization.

Figure 5b illustrates the evolution of the number-average molecular weight (Mn) over time, comparing the theoretical values ($M_{n,theo}$., black squares) and experimental values obtained from GPC ($M_{n,GPC}$, red circles). The strong correlation between the theoretical and experimental M_n values indicates a well-controlled polymerization process, with molecular weight increasing linearly with reaction time. This linear trend supports a living polymerization mechanism, where the degree of polymerization is directly proportional to monomer conversion.

The lower panel of Figure 5b presents the dispersity ($Đ=M_w/M_n$) values, which remain consistently low ($Đ<1.22$) throughout the polymerization. This narrow molecular weight distribution further supports the controlled nature of the polymerization, minimizing termination and chain transfer reactions. The stability of Đ at low values suggests a uniform chain growth, reinforcing the controlled/living character of the polymerization process.

The peak broadening remains minimal, and all GPC traces exhibit a unimodal distribution, further supporting the formation of well-defined polymer chains with low dispersity. The increasing molecular weight with time aligns well with the kinetic analysis presented in Figure 6, reinforcing that the polymerization follows pseudo-first-order kinetics with respect to monomer consumption.

The final retention time plateau at later stages (T_{150} to T_{210}) suggests that monomer conversion is

nearing completion. These results demonstrate that PEtOx polymerization proceeds in a controlled and

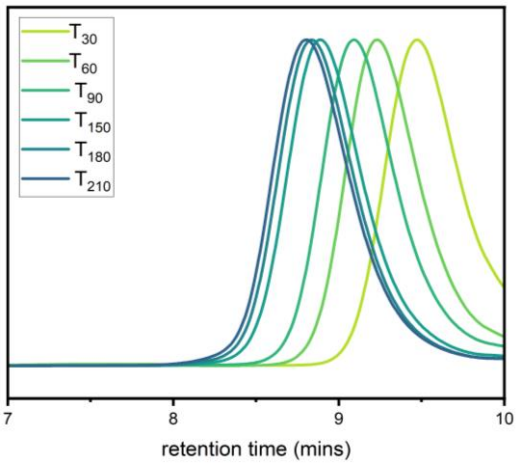

Figure 6
GPC traces of PEtOx after purification using THF with 2% TEA as an eluent

predictable manner, making it suitable for well-defined polymer synthesis. The numerical data summarizing these molecular weight and dispersity values at different time points are presented in Table 1 for clarity and reference.

Table 1
Number-average molecular weight (Mn), weight-average molecular weight (Mw), and dispersity index (Đ = Mw/Mn) of the samples obtained at different polymerization times.

Time	Mn (Da)	Mw (Da)	Đ=M_w/M_n
T_{30}	1000	1100	1.1
T_{60}	1400	1600	1.14
T_{90}	1600	1900	1.16
T_{150}	2000	2400	1.19
T_{180}	2400	2900	1.21
T_{210}	2600	3000	1.2

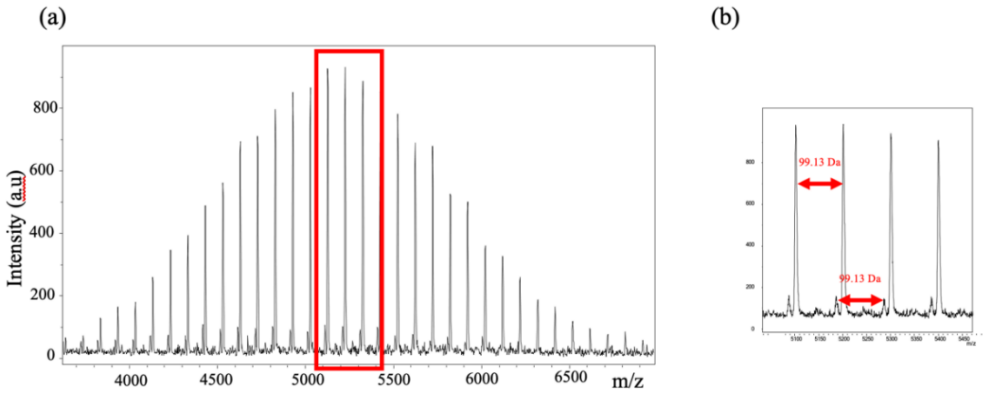

Figure 7
MALDI-ToF spectra for AdTos initiated polymerization of EtOx at 120℃. (a) Full spectrum, (b) magnified inset. [M + K+]calc = 5108.73 Da, [M + K+]found = 5108.19 Da.

Figure 7 presents the matrix-assisted laser desorption/ionization time-of-flight (MALDI-ToF) mass spectrum of poly(2-ethyl-2-oxazoline) (PEtOx), confirming the well-defined polymer structure. The primary spectrum (Figure 7a) exhibits a series of evenly spaced peaks, corresponding to different polymer chain lengths with a consistent repeating unit. The observed molecular weight distribution aligns well with GPC data from Figure 6, further supporting the controlled nature of the polymerization process.

The zoomed-in inset (Figure 7b) highlights the isotopic resolution of the polymer peaks, with a clear peak-to-peak separation of 99.13 Da, which corresponds to the molecular weight of a single 2-ethyl-2-oxazoline (EtOx) repeat unit. Additionally, a minor distribution (highlighted in the zoomed-in region) is observed, which corresponds to the hydrogen adduct [28].

CONCLUSION

In this study, the kinetic aspects of poly(2-ethyl-2-oxazoline) (PEtOx) polymerization initiated by 2-adamantyl tosylate (AdTos) were systematically investigated. The results demonstrated that the polymerization follows pseudo-first-order kinetics, with a strong linear correlation between monomer conversion and time, confirming a controlled polymerization process. The molecular weight evolution exhibited a close agreement between theoretical and experimental values, further supporting the living nature of the polymerization with minimal chain transfer and termination reactions.

Gel permeation chromatography (GPC) analysis confirmed a narrow molecular weight distribution, with dispersity (Đ) remaining below 1.22 throughout the reaction, reinforcing the controlled/living character of the polymerization. Additionally, matrix-assisted laser desorption/ionization time-of-flight (MALDI-ToF) mass spectrometry validated the formation of well-defined polymer chains with precise end-group fidelity.

These findings contribute to a deeper understanding of the cationic ring-opening polymerization (CROP) of 2-oxazoline monomers and demonstrate the efficiency of AdTos as an initiator for achieving well-defined PEtOx structures. The insights gained from this study provide a foundation for the rational design of tailored polyoxazoline architectures, which can be further explored for advanced material applications. Future research may extend this approach to other oxazoline monomers and investigate the impact of reaction parameters on polymer microstructure and functionality.

Ethical Statement
The present study is an original research article designed and produced by the authors.

Author Contributions
Research Design (CRediT 1) S.K. (%40) – G.Y. (%30) – R.B. (%30)
Data Collection (CRediT 2): S.K. (%40) – G.Y. (%30) – R.B. (%30)
Research – Data Analysis – Validation (CRediT 3-4-6-11): S.K. (%40) – G.Y. (%30) – R.B. (%30)
Manuscript Writing (CRediT 12-13): S.K. (%40) – G.Y. (%30) – R.B. (%30)
Text Revision and Improvement (CRediT 14): S.K. (%30) – G.Y. (%30) – R.B. (%40)

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Conflict of Interest
The authors declare that there is no conflict of interest regarding the publication of this paper.

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