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Chemical Modifications of Polymer for Improved Anti-Microbial Action

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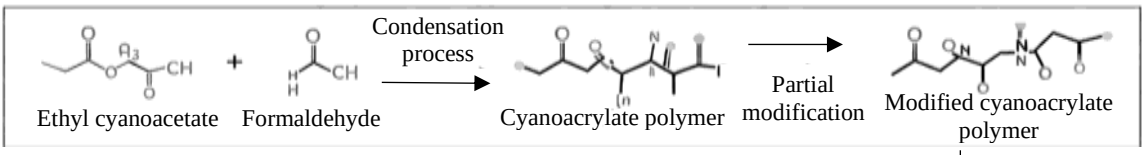
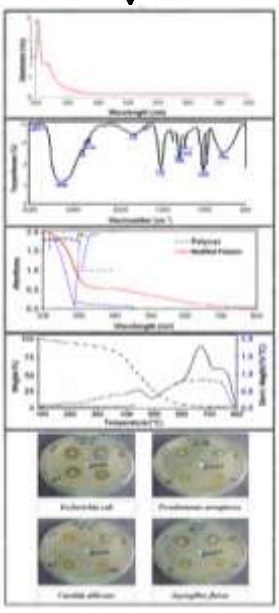
Abstract

Infections caused by pathogenic microorganisms are a significant concern. Polymers, large molecules formed by chemically connecting monomers, have acquired significant interest due to their intrinsic properties. Polymers function as a matrix for the components that contain antibacterial agents. Developing polymers with antibacterial activity is a crucial research area that aims to alleviate the problem of microbe contamination. The antibacterial polymers typically exhibit prolonged effectiveness. Modification of polymers enhances their anti-microbial properties. Cyanoacrylate polymers hold notable importance because of their strong adhesive nature, rapid polymerization, and potential for structural customization. In this present investigation, the cyanoacrylate polymer can be synthesized using ethyl cyanoacetate monomer and formaldehyde via the condensation method. Further, the synthesized cyanoacrylate polymer was modified partially. The partially modified cyanoacrylate polymer was characterised by Ultraviolet-visible spectroscopy, Fourier transform infrared spectroscopy, Nuclear magnetic resonance, Thermo gravimetry analysis, Differential scanning calorimetry and Gel permeation chromatography. Spectroscopic and thermal analyses confirmed successful modification and revealed improved thermal stability and structural integrity. The antimicrobial efficacy of the partially modified cyanoacrylate polymer was assessed against selectedGram-positive, Gram-negative, and fungal strains. The antimicrobial activity of a partially modified cyanoacrylate polymer showed a superior level of inhibitory action against microorganisms. These findings emphasize the potential of modified cyanoacrylate polymers as multifunctional materials for biomedical and environmental applications, offering prolonged and reliable antibacterial performance.

Graphical abstract





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Keywords: Anti-microbial property, cyanoacrylate, FTIR, NMR, polymer, UV-visible

INTRODUCTION

Polymers are enormous molecules composed of many small, repeating units known as monomers. The process of combining monomers to produce polymers is known as polymerization [1]. Antimicrobial polymers are materials designed to kill or limit the growth of bacteria on the surface or in the environment that surrounds them. Quaternary nitrogen compounds possess inherent antibacterial action, and polymers can serve as a backbone that enables very small biocides to demonstrate their activity [2]. Antimicrobial polymers currently under research include substituted modified natural polymers, polymers with multiple units linked to their backbone, polymers which have a terminal satellite biocide unit, and polymer-inorganic antimicrobial composites as well. These are polymers that have little antibacterial effect by themselves, yet can be enhanced by incorporating specific groups which generate antimicrobial activity [3].

Cyanoacrylates are a type of acrylic monomers that induce rapid polymerization in presence of water via an anionic or zwitterionic polymerization process. These compounds are formed by combining ethyl cyanoacrylate and its derivatives. The cyanoacrylate functional group present in the monomer undergoes polymerization quickly in H_2O, forming lengthy and excellent chains [4]. Cyanoacrylate ester is a colorless and monomeric liquid that is available commercially. It rapidly forms a polymer at room temperature following an anionic reaction with weak bases. The initiation of polymerization occurs through anionic species [5].

SYNTHESIS OF CYANOACRYLATE POLYMER

Ethyl cyanoacetate was placed in a round-bottom flask with benzene. Paraformaldehyde powder was introduced and the flask was placed in an oil bath. Pyridine was added, and the contents were stirred and heated to boil. The water generated during the reaction was removed azeotropically [6]. The reaction was allowed to proceed until the estimated amount of water was removed. After letting the reaction mixture settle to cool to room temperature, the contents were rinsed with water, dilute hydrochloric acid and then with water (to remove pyridine, NaOH, and CH_2O). Upon removal of the solvent benzene, an amber-color resinous product was formed, which upon standing gave dry solid-ethyl cyanoacrylate polymer which has a yield. The synthesis process of the polymer is shown in Figure 2.

Modification of Cyanoacrylate Polymer

Ethyl cyanoacrylate polymer was dissolved in THF and the mixture was stirred using a magnetic stirrer. Hydrazine hydrate was added, and stirring was continued overnight at 80°C on an oil bath. A pale-yellow precipitate was formed, which was separated by decanting the solvent. The resulting precipitate was washed repeatedly with THF to eliminate any unreacted starting material. The yellow precipitate obtained was water soluble and had a yield [7]. The modification steps are represented in Figure 3, while the overall reaction scheme is presented in Figure 4.

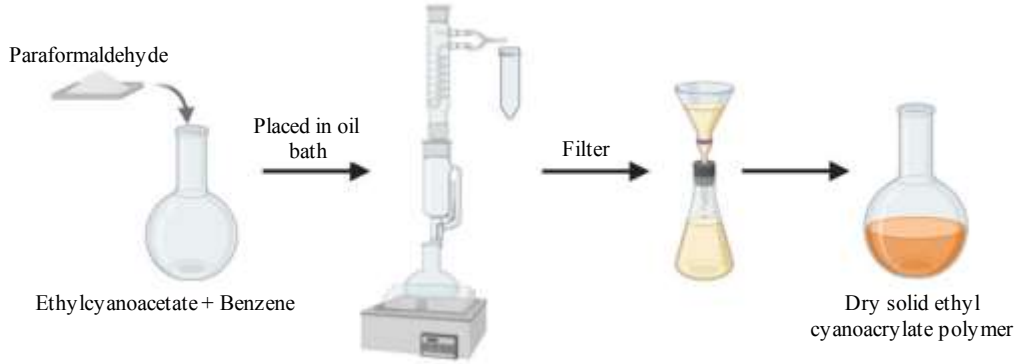


Figure 2. Synthesis of partially cyanoacrylate polymer.

Characterization Techniques

Several analytical methods characterized the partially modified cyanoacrylate polymer. UV-Vis spectroscopy detected chromophores and electronic transitions via absorption spectra. FTIR identified functional groups through molecular vibrations using a Perkin Elmer instrument with KBr pellets [8]. TGA assessed thermal stability by mass changes. NMR elucidated atomic-level structure in DMSO-d_6 with TMS reference on JEOL equipment. DSC explored phase transitions and glass transition, while GPC determined molecular weight distribution in organic solvents [9].

Antimicrobial Activity

The polymer sample demonstrated antibacterial and antifungal properties against various microorganisms, specifically one gram-positive bacterial strain and one gram-negative bacterial strain. Additionally, they examined its effects on the fungal cultures. The bacterial and fungal strains were obtained from reputable institutions in India, ensuring the reliability of the study [10].

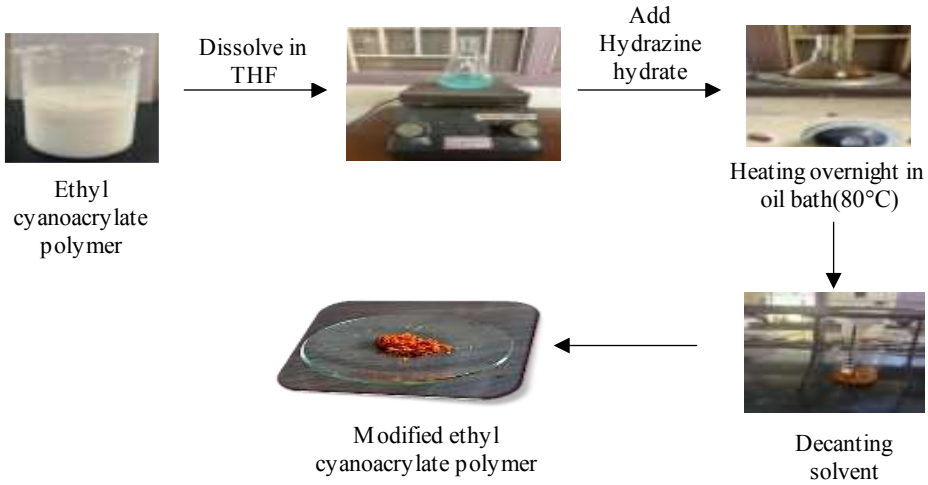


Figure 3. Modification of cyanoacrylate polymer.

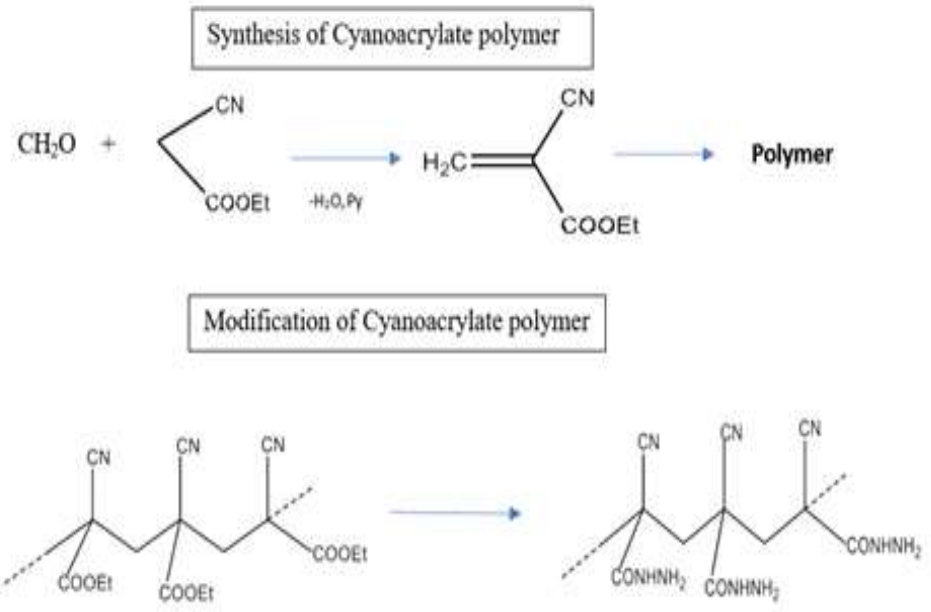


Figure 4. Scheme of synthesis and modification of partially cyanoacrylate polymer.

The antibacterial and antifungal activities of the modified cyanoacrylate polymer were evaluated using the disc diffusion method. Sterilized Mueller–Hinton agar medium was prepared and inoculated with the respective test organisms. For antibacterial assessment, the bacterial strain was placed uniformly onto the agar plates, while for antifungal evaluation, the fungal strain was similarly introduced [11]. The modified polymer samples were prepared at concentrations of 60, 80, and 100 µg/mL and applied to the discs. The filter paper disc consists of 5 µg of amoxicillin that was utilized as a positive control for antibacterial activity, whereas a disc impregnated with 5 µg of fluconazole served as the positive control for antifungal activity. Every plate was evaluated by measuring the zones of inhibition following a 24-hour incubation period at 37°C to assess antimicrobial efficacy. The antibacterial study was repeated to ensure reproducibility and validation of the results.

RESULT AND DISCUSSION

UV-Visible Spectroscopy

The UV–Vis spectrum of modified cyanoacrylate polymer is shown in Figure 5 that exhibits intense absorption in the ultraviolet region reaching a maximum absorption at 244nm. This peak can be assigned to $\pi \rightarrow \pi^*$ transitions of the chromophoric groups. The presence of this intense peak confirms the retention of the unsaturated polymer's structure after chemical modification. The absence of significant absorption in the visible region indicates that no extended conjugation was introduced during modification. Consequently, the polymer maintains good optical transparency, making it suitable for optically clear applications [12].

Fourier-Transform Infrared Spectroscopy

The FT-IR spectrum of the modified cyanoacrylate polymer confirms the preservation of the polymer backbone along with the incorporation of new functional groups after modification. The FTIR spectrum of the modified polymer is presented in Figure 6. A characteristic absorption band observed at 2127.96 cm^{-1} represents the nitrile (–C≡N) stretching vibration, indicating the retention of the cyanoacrylate structure. The occurrence of a band at 1648.48 cm^{-1} is attributed to amide carbonyl (C=O) stretching, suggesting the successful introduction of nitrogen-containing functionalities. A broad absorption centered around 3427.52 cm^{-1} is assigned to N–H stretching vibrations, further supporting an incorporation of amine or amide groups during the modification process. The band at 2918.79 cm^{-1} corresponds to aliphatic C–H stretching. Additional absorptions in the region near 1410.51 cm^{-1} are related with C–C or C–H bending vibrations, whereas the peak at 1020.57 cm^{-1} is characteristic of C–N stretching, indicating the presence of nitrogen-containing substituents. A slight shift of the carbonyl band toward lower wavenumbers along with the broadening of the N–H band suggests the formation of intermolecular hydrogen bonding within the modified polymer [13]. The characteristic FTIR absorption peaks of the partially modified cyanoacrylate polymer are presented in Table 1.

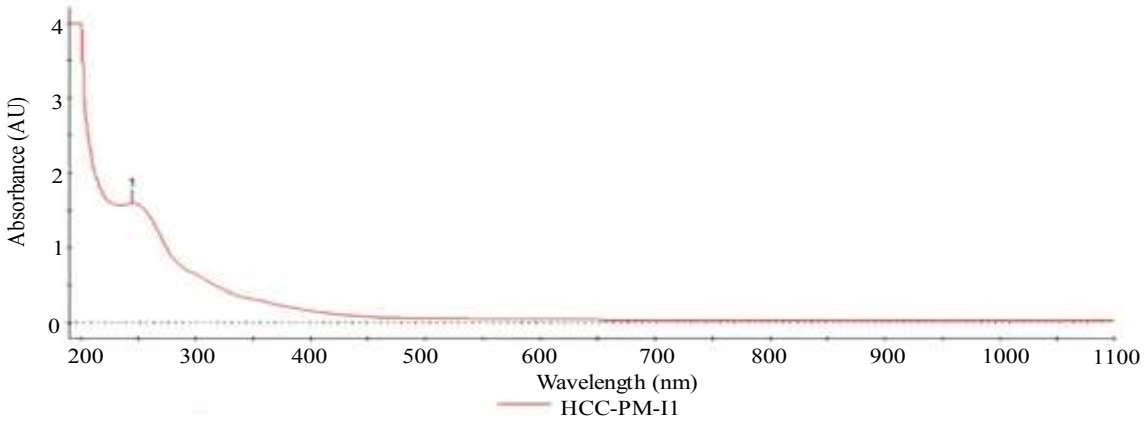


Figure 5. UV Visible spectrum of partially modified cyanoacrylate polymer

Table 1. FTIR peaks of partially modified cyanoacrylate polymer.

Frequency	Band	Functional group
3936.91 cm^{-1}	O-H stretching	Hydroxyl
3427.52 cm^{-1}	N-H stretching	Amine
3008.01 cm^{-1}	=C-H stretching	Alkene
2918.79 cm^{-1}	C-H stretching	Aliphatic
2601.23 cm^{-1}	O-H stretching	Carboxylic acid
2127.98 cm^{-1}	C=N stretching	Nitrile
1648.48 cm^{-1}	C=C stretching	Amide
1436.31 cm^{-1}	-CH_2 bending	Alkene
1317.40 cm^{-1}	C-N stretching	Amine
1025.57 cm^{-1}	C-O-C stretching	Ester

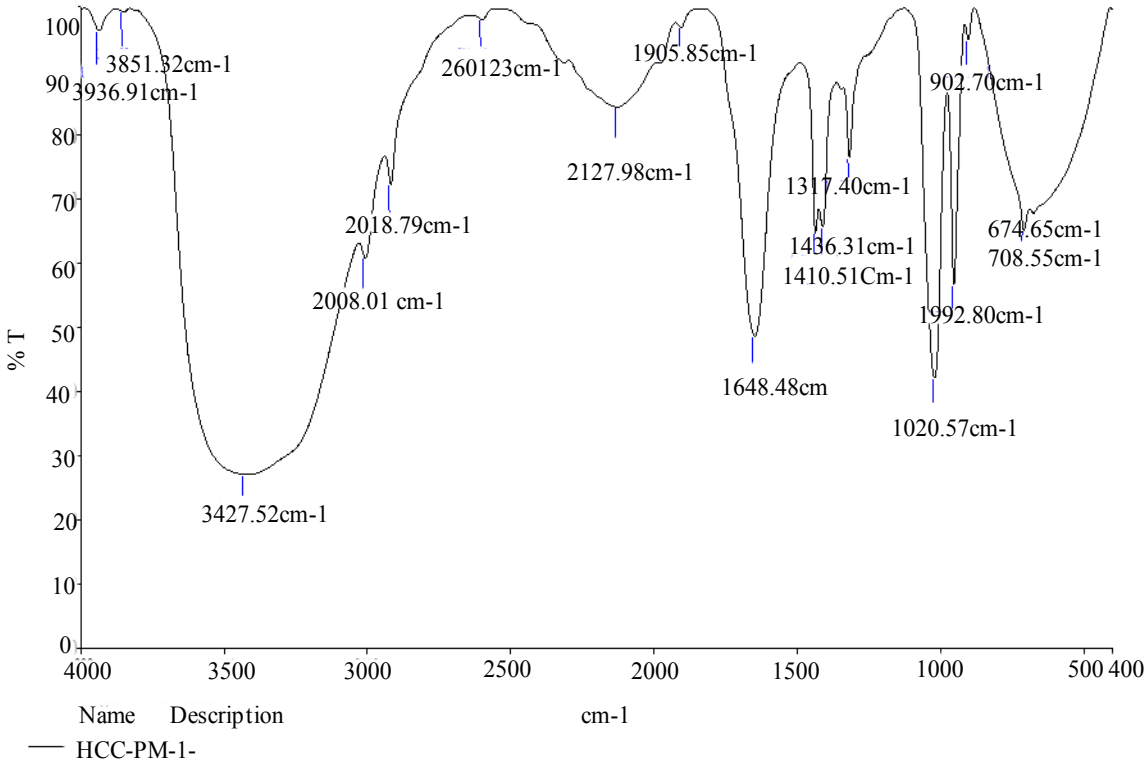


Figure 6. FTIR spectra of partially modified cyanoacrylate polymer.

Nuclear Magnetic Resonance Spectroscopy

The ^1H NMR spectrum provides important information regarding the proton environment in the partially modified cyanoacrylate polymer shown in Figure 7. The signals observed in the δ 1.0–1.5 ppm region are attributed to aliphatic methyl (–CH_3) protons present in the polymer chain. Resonances in the δ 2.5–3.0 ppm range correspond to methylene or methine protons located adjacent to nitrogen atoms (–CH_2–N or –CH–N), indicating the incorporation of amine-containing groups during the modification process. Signals appearing in the δ 3.0–4.0 ppm region are assigned to protons attached to carbon atoms bonded to electronegative atoms, such as nitrogen or oxygen (–CH–N or –CH–O). Broad and weak signals within this region may also arise from exchangeable N–H protons. The presence of these new proton environments, when compared with the unmodified polymer, confirms the successful partial functionalization of the cyanoacrylate polymer [14].

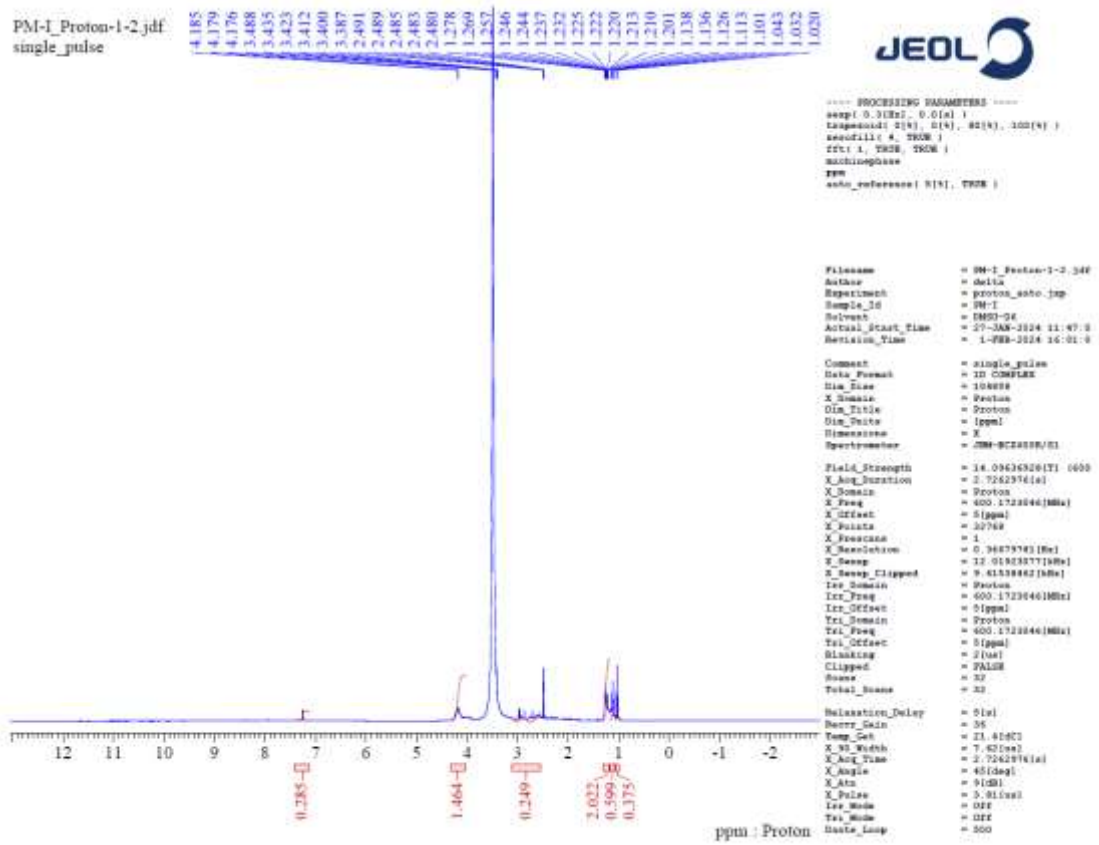


Figure 7. NMR spectra of partially modified cyanoacrylate polymer.

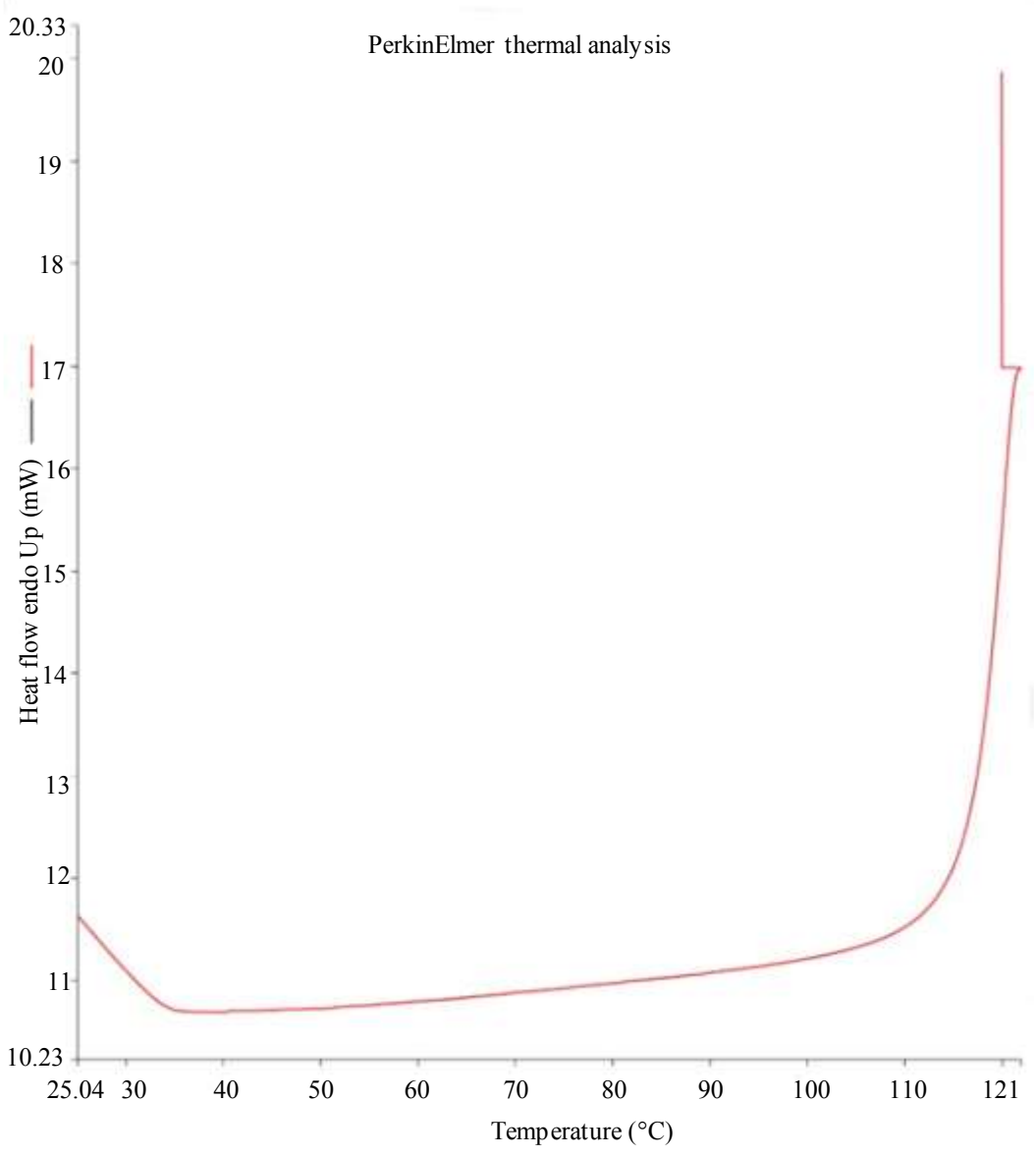
Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) analysis was performed to find stability and thermal transitions of the partially modified polymer. The DSC thermogram is illustrated in Figure 8. A small endothermic peak was found in the temperature range of 30–45°C, which can be attributed to the evaporation of physically trapped water or the softening of the polymer. A sharp endothermic peak was found in the temperature range of 115–120°C, that can be assigned to the melting of crystalline regions or softening of polymer. Difference in this transition shows that the distribution of crystalline phases is relatively homogeneous. No additional melting and crystallization peaks were recorded in the second heating scan, and it is likely that the polymer mostly exhibits amorphous characteristics after the first thermal treatment and does not undergo cold crystallization during the reheating process. It was observed that a substantial thermal event occurred in the band 290–300°C which showed the onset of the thermal degradation of the polymer. The absence of some of the transitions and high temperature of degradation shows uniformity in the structure, high intermolecular forces, and good thermal stability of the product. DSC analysis shows that the partially modified polymer formed has a moderate thermal stability and that it is structurally stable even in the temperature up to approximately 280°C [15].

Thermogravimetric Analysis

The thermogravimetric and derivative thermogravimetric curves give specific understanding of the thermal degradation behavior and stability of the synthesized polymer in the temperature range of 30–400°C. The thermal degradation behavior is shown in Figure 9. The polymer has a three-stage degradation cycle in which the first stage starts at an onset temperature of 42.24°C and stops at 97.99°C signifying that there is a mass-loss at an early stage. The TGA curve indicates a sharp decline in this area which can be attributed to a significant weight loss of 51.96%. The DTG curve shows a clear peak at a temperature of 81.88°C, and the maximum rate of degradation is -12.888%/min, which proves it is

the major mass-loss step. This can be understood by the elimination of moisture that has been physically adsorbed, remaining solvent molecules' evaporation, loss of low-molecular-weight oligomers or unreacted monomers, breakdown of functional groups that are thermally labile. The steep and sharp DTG peak indicates that volatile molecules are loosely coupled to the polymer network. After initial degradation, the polymer steadily loses its mass at a slow rate with a lower slope in the TGA curve. This phase presents a high level of structural stability, as compared to the initial stage. The lack of high DTG peaks is an indication of partial degradation of functional substituents. Here, it is the degradation of relatively stable chemical bonds in the modified polymer. The last stage of decomposition at above 250°C, the polymer undergoes a continuous yet lower rate of mass loss, which corresponds to the breakdown of the main polymer chain, structural rearrangement and formation of carbonaceous residue (char). When the temperature reaches 400°C, the mass left is about 16–18%, which means that char was produced and partially carbonized. The existence of residue indicates that the substance has some level of crosslinking or aromatic nature which increases thermal resistance when the temperature is high [16]. This thermal behavior provides crucial information for assessing the polymer's processing limits and possible high-temperature applications, as well as confirming the impact of chemical modification on the polymer structure.



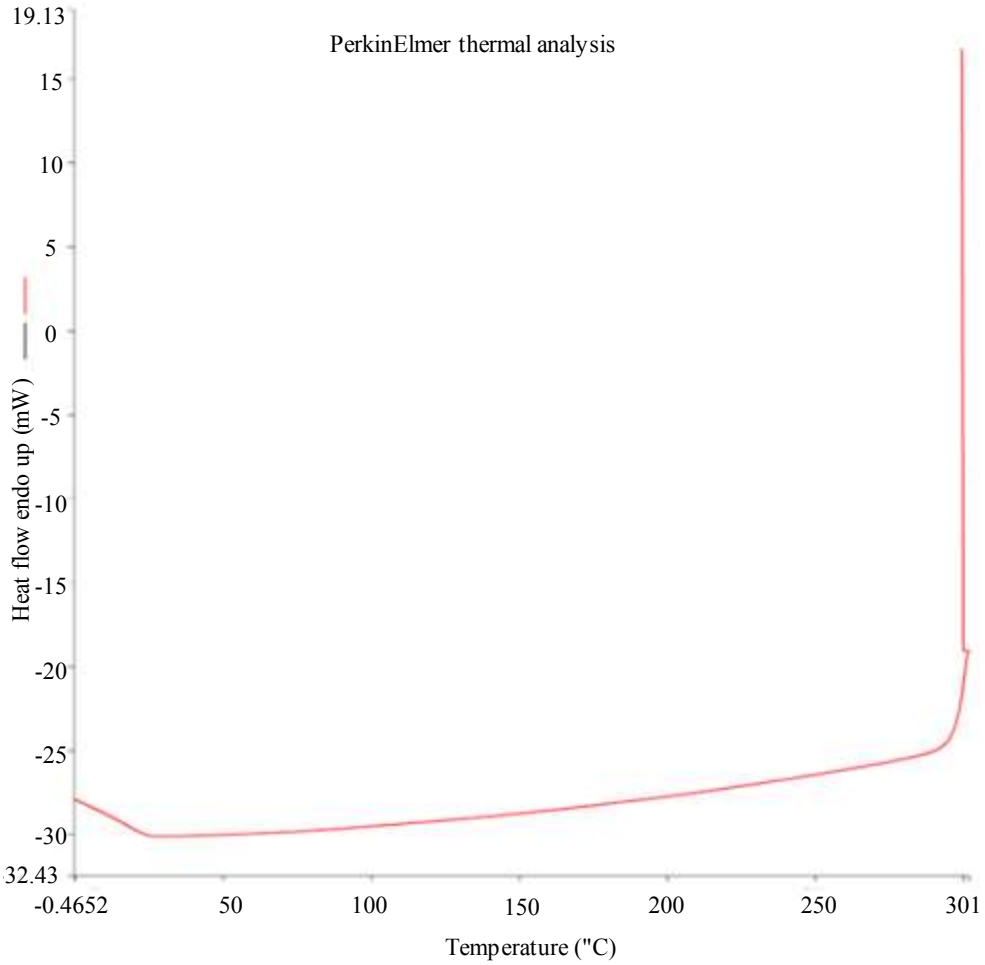


Figure 8. DSC curves of partially modified cyanoacrylate polymer.

Gel Permeation Chromatography

Gel Permeation Chromatography (GPC) analysis of partially modified cyanoacrylate polymer was performed using a Shimadzu LC system with a third-order polynomial calibration curve showing excellent linearity ($R^2 = 0.9999925$), confirming high reliability of molecular weight determination. The molecular weight distribution curve is presented in Figure 10. The chromatogram reveals a clear bimodal molecular weight distribution, indicating the presence of two distinct polymer populations at retention time of 7.724 min and 9.557 min. The previous renouncing peak 1 with 9.78% of the total area was associated with the larger molecular weight fraction and had Mn = 9823 g/mol, Mw = 11294 g/mol, and a small polydispersity index (PDI, Mw/Mn) of 1.15, indicating a rather homogenous distribution of chain lengths in this fraction. On the other hand, the later eluting peak 2 with 90.22% of the total area was mostly low molecular weight oligomeric species with Mn = 509 g/mol, Mw = 550 g/mol and a very narrow PDI of 1.08, indicating controlled but limited chain growth. The molecular weight characteristics of the modified polymer in general and in particular were Mn = 566 g/mol and Mw = 1693 g/mol with broad PDI = 2.99 which indicated the presence of two distinct populations of the polymer. The large difference between Mn and Mw confirms that despite the large number of the oligomeric fraction, the small proportion of high molecular weight fraction has a substantial impact on the weight-average molecular weight. The bimodal distribution shows incomplete polymerization or chain transfer and premature termination reactions during the synthesis which leads to the formation of more oligomers with little or no propagation up to higher molecular weights. This heterogeneity in molecular weight distribution can have a profound effect on the physicochemical properties of polymer which can include viscosity, mechanical strength, film forming capacity and thermal stability [17]. The

molecular weight of the partially modified cyanoacrylate polymer was determined and the results are summarized in Table 2.

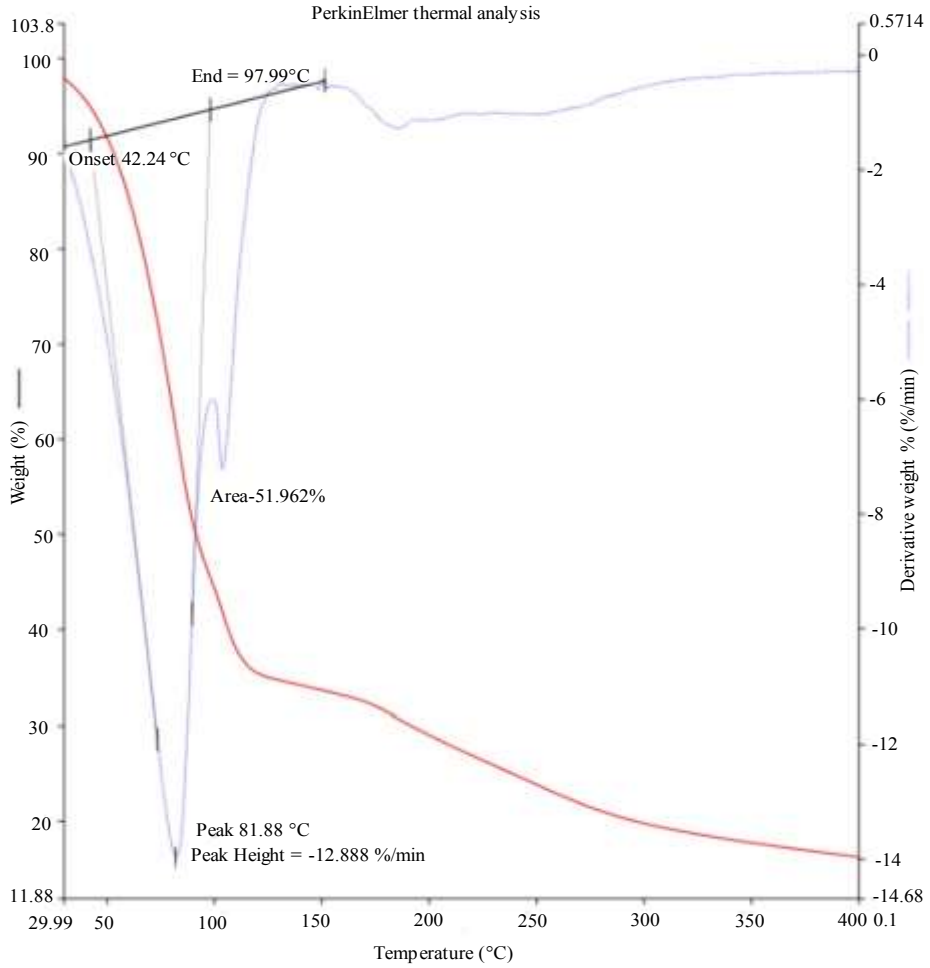


Figure 9. TGA of partially modified cyanoacrylate polymer.

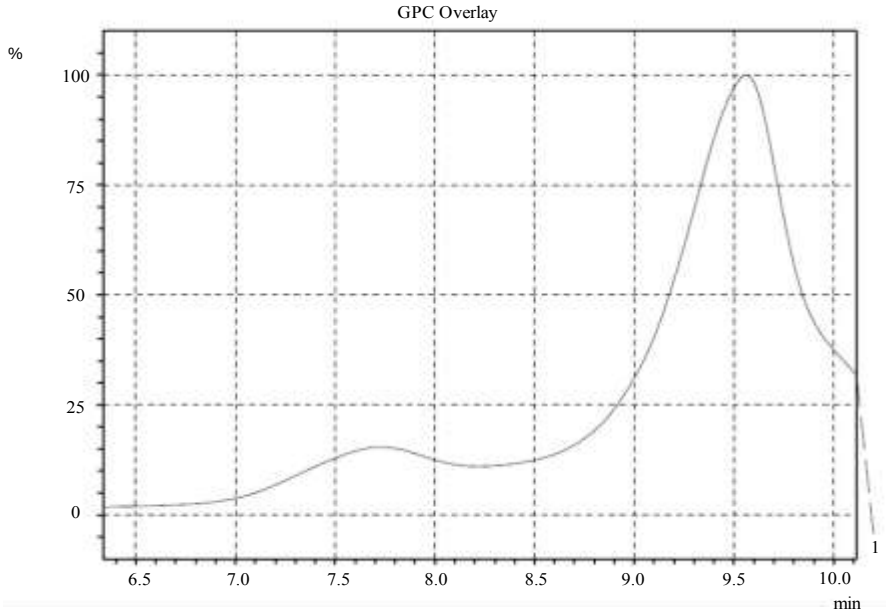


Figure 10. GPC curve of the partially modified cyanoacrylate polymer.

Table 2. Molecular weight determination of partially modified cyanoacrylate polymer.

S.N.	Time(min)	Molecular weight
1	6.548	70000
2	6.749	50000
3	7.048	30000
4	7.654	10000
5	8.056	5000
6	9.017	1000

Anti-Bacterial Activity

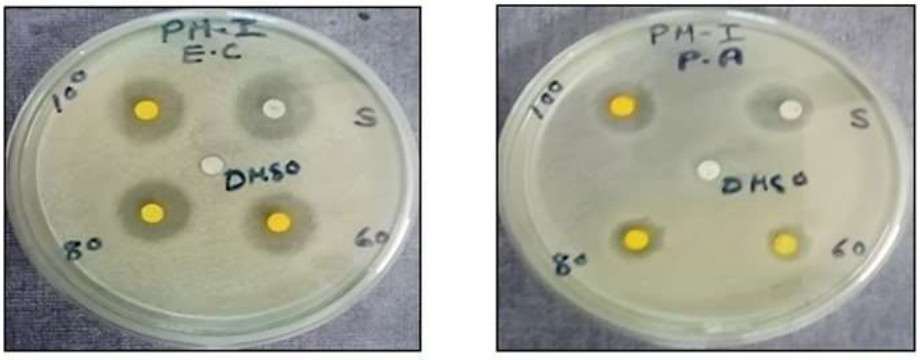
The cyanoacrylate polymer has been modified partially resulting in antibacterial activity. The antimicrobial activity is visually represented in Figure 11. Table 3 indicates the inhibitory action of the modified polymer against various bacterial strains. The inhibitory action to microorganisms' growth at various concentrations of 60, 80, 100 µg/mL is given in Table 3. Amoxicillin is used as a standard. The mean zone of inhibition obtained by modified cyanoacrylate polymer for the tested specifically for the bacterial strains 'Escherichia coli' and 'Pseudomonas aeruginosa'. The maximum suppression was spotted at 100 µg/mL for Escherichia coli in comparison with other bacteria. Hence from the result obtained, it is clear that the partially modified cyanoacrylate polymer has maximum level of anti-bacterial activity. Methanol control did not exhibit any inhibition (0 mm), which proved that the resulted antibacterial effect was due to the sample and not to the solvent [18].

Anti-Fungal Activity

The antifungal activity was performed for partially modified cyanoacrylate polymer. The antifungal activity is shown in Figure 12. Table 4 indicates the inhibitory action of modified polymer against various fungal strains. The inhibitory action to microorganisms' growth at various concentrations of 60, 80, 100 µg/mL is given in Table 4. Fluconazole is used as a standard. The mean zone of inhibition obtained by modified cyanoacrylate polymer for tested against Candida albicans and Aspergillus flavus [19]. The maximum inhibition was observed at 100 µg/mL for Candida albicans in comparison with Aspergillus flavus. DMSO (solvent control) had no inhibition as this implies the antifungal effect was not caused by the solvent but only by the sample. Hence from the result obtained, it is clear that the partially modified cyanoacrylate polymer has antifungal activity at the maximum level [20].

Table 3. In Vitro Antibacterial activity of partially modified cyanoacrylate polymer.

Samples	Concentrations (µg/ml)	Organisms/Zone of inhibition (mm)	
		Escherichia coli	*Pseudomonas aeruginosa*
Samples	60	5	3
	80	6	5
	100	9	7
Standard (Std) (Amoxicillin)	10 µl/disc	10	9

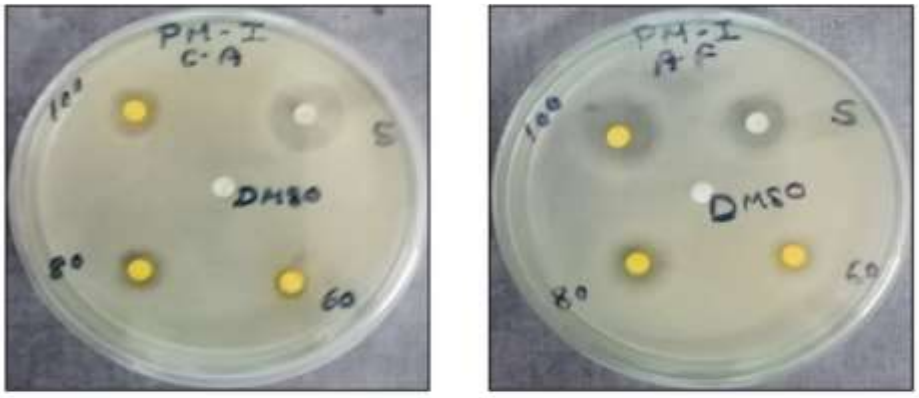


Escherichia coli Pseudomonas aeruginosa

Figure 11. Antimicrobial activity of partially modified cyanoacrylate polymer.

Table 4. In Vitro Antifungal activity of partially modified cyanoacrylate polymer.

Samples	Concentrations (µg/ml)	Organisms/zone of inhibition (mm)	
		Candida albicans	*Aspergillus flavus*
Sample	60	2	0
	80	4	3
	100	6	5
Standard (Std) (Fluconazole)	10 µl/disc	8	6
DMSO	10 µl/disc	0	0



Candida albicans Aspergillus flavus

Figure 12. Antifungal activity of partially modified cyanoacrylate polymer.

CONCLUSION

The cyanoacrylate polymer was successfully synthesized through a condensation reaction between formaldehyde and cyanoacetate, yielding an amber-colored product characteristic of the unsubstituted polymer. Chemical modification was achieved efficiently, as visually confirmed by the formation of a pale-yellow precipitate, a clear indicator of structural alteration. UV-Vis spectroscopy revealed a notable absorption peak at 244nm in the modified polymer, attributable to newly introduced conjugated systems or chromophores from the modifying groups. FTIR spectra shows emergence of multiple new absorption bands corresponding to key functional groups, such as amines, carbonyls, or other heteroatoms.

^{1}H-NMR analysis provided definitive structural proof, with diagnostic signals at δ 1.0–1.5 ppm (methyl protons, $–CH_3$), δ 2.5–3.0 ppm (protons on carbons α to amines, $–CH_2–NH–$), and δ 3.0–4.0 ppm (NH_2-bound protons). These shifts and integrations confirm the successful grafting of amine-containing moieties onto the cyanoacrylate backbone. TGA and DSC analysis confirmed enhanced thermal stability, featuring higher decomposition onset temperatures and sharper thermal transitions revealing the improved cross-linking or rigidity. Gel permeation chromatography (GPC) quantified the molecular weight distribution, showing a controlled increase in number-average molecular weight and weight-average molecular weight alongside a refined polydispersity index.

Antimicrobial polymers inhibit microbial growth by disrupting cell adhesion, membrane integrity, or metabolism, typically via bioactive groups like quaternary ammonium or imidazolium salts. These results underscore its potential in biomedical coatings, environmental applications and antimicrobial surfaces. This study affirms the modification strategy's success, establishing this polymer as a versatile material for advanced applications.

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