

Synthesis and characterization of inclusion complexes of aliphatic-aromatic poly(Schiff base)s with β -cyclodextrin (highlight)

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Abstract This paper describes the formation of polymer inclusion complexes (polymer-CD-ICs) between β -cyclodextrin (β -CD) and aliphatic-aromatic poly(Schiff base)s. Fourier transform infrared (FTIR) spectroscopy, ^1H nuclear magnetic resonance spectroscopy (^1H -NMR), thermogravimetric analysis (TGA) and X-ray diffraction (XRD) have been used to observe the formation of polymer-CD-ICs. In FTIR spectra, the characteristic peaks of β -CD at 3391 cm^{-1} shifted to 3418 cm^{-1} and the intense peak at 1602 cm^{-1} due to the $-\text{C}=\text{N}-$ stretching vibration diminished after formation of inclusion complexes. Compared the ^1H -NMR of polymer-CD-ICs with β -CD, the chemical shift of the protons H-3, H-5 have shifted to higher field after the formation of inclusion complexes, which is perhaps due to the interaction of these protons with polymers. The TGA analysis revealed that the polymer-CD-ICs had better thermal stability than β -CD, suggesting that the polymer increased the stability of β -CD. The X-ray diffraction patterns displayed that the strong peak for both polymer-CD-ICs at approximately 20.0° (2θ) may confirm their IC formation.

Keywords Cyclodextrin · Poly(Schiff base)s · Inclusion complexes

Introduction

Cyclodextrins (CDs) are cyclic oligosaccharides with D-(+)-glucose as the six to eight repeating unit coupled by α -1,4-linkages. They are called α -, β -, and γ -cyclodextrin, respectively. They have been extensively studied in supramolecular chemistry as host molecules. Since CDs were discovered, a large many inclusion complexes of CDs with various low molecular weight compounds have been prepared and characterized [1]. The major driving forces of the formation of CD inclusion compounds are hydrophobic and van der Waals interactions between the inner surface of the CD ring and the hydrophobic sites on the guest [2]. Over the past decade, there has been a steady growth of interest in the synthesis and application of polymer-CD-ICs. Since Harada's group [3] first found that the α -CD and poly(ethylene glycol) (PEG) formed inclusion complex, a great number of researches on inclusion complexes of CDs and various kinds of polymer were reported. Subsequently, Harada's group discovered successively that many polymers were capable of forming inclusion complexes with different cyclodextrins, such as: α -CD was also suited to include poly(oxytrimethylene) and poly(oxytetramethylene) [4], poly(propylene oxide) formed insoluble inclusion complexes with β -CD [5], γ -CD was able to accommodate poly(methylvinyl ether) [6], aliphatic poly(adipates) form inclusion compounds with α -CD and γ -CD [7], polyethylene and α -CD formed inclusion compounds [8], poly(dimethylsiloxane) (PDMS) was included by γ -CD [9]. In addition, Jiao [10] prepared the inclusion compounds of oligo(tetrafluoroethylene oxide-co-difluoromethylene) and oligo(hexafluoropropylene oxide). Huang [11] reported that nylon-6 and α -CD formed inclusion complex. Yamaguchi [12] prepared the polyurethane CD inclusion compounds by polyaddition of linear aliphatic diols and

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methylenediphenyl-4, 4'-diisocyanate(MDI) in the presence of permethylated α - or β -CD in DMF solution. Yoshida [13] found that polyaniline was included by β -CD in N-methylpyrrolidone/water mixtures and leading to a blue precipitate at temperatures below 2 °C.

Poly(Schiff base)s are a kind of particularly attractive polymers due to their interesting properties such as: good mechanical strength [14], attractive thermal stability [15], nonlinear optical [16] and liquid crystal [17]. However, poor solubility in common organic solvents and difficult to liquate limited poly(Schiff base)s' practical applications in various fields. Thus many groups have synthesized soluble poly(Schiff base)s through introducing alkyl or alkoxy chains into the polymers or inserting solubility enhancing groups in the backbone [18]. In view of these and in continuation of our previous work on the supramolecular chemistry [19–21], we synthesized and characterized of some aliphatic-aromatic poly(Schiff base)s obtained by polycondensation reactions of 1, 4-phenylenediamine with some aliphatic-aromatic dialdehydes. At the same time, we prepared the inclusion complexes of aliphatic-aromatic poly(Schiff base)s and β -CD via heating techniques. Fourier transform infrared(FTIR) spectroscopy, ^1H nuclear magnetic resonance spectroscopy(^1H -NMR), thermogravimetric analysis(TGA) and X-ray diffraction(XRD) are used to characterize the inclusion complexes. Owing to the interesting properties of linear aliphatic-aromatic poly(-Schiff base)s, it is interesting to introduce them to cyclodextrins.

The scope of this preliminary paper is to present the synthesis of the inclusion complexes based on aliphatic-aromatic poly(Schiff base)s as linear polymer and β -cyclodextrin as macrocyclic compound. The next step in our studies is to analyze the structure of polymer-CD-ICs in detail and study electro-optical properties of this kind of inclusion complexes.

Experimental

Materials

β -CD (product of Shanghai Reagent Factory, China) was recrystallized twice from distilled water and dried in vacuum prior to use. 1, 4-phenylenediamine, *p*-hydroxybenzaldehyde and α , ω -dibromoalkane were products from Shanghai Reagent Factory(China), while the dialdehydes were synthesized according to the literature data [22]. The monomers

were purified by recrystallization. The solvents (N, N-dimethylformamide (DMF), N-methyl-pyrrolidone (NMP) and absolute alcohol) were used as received.

Measurements

FTIR spectra were measured using a Nexus670X FTIR spectrometer at room temperature in the range between 4000 cm^{-1} and 400 cm^{-1} , with a resolution of 2 cm^{-1} and 20 scans. Powder samples were prepared by dispersing the samples in KBr and compressing the mixture to form disks.

^1H NMR spectra were obtained as solution using a Mercury Plus-400 Varian instrument at room temperature with DMSO- d_6 and D $_2$ O solvent and tetramethylsilane (TMS) as internal standard.

Thermogravimetric analysis (TGA) of samples was made using DT-40 thermogravimetric analyzer at a heating rate of 10 °C/min with nitrogen used as purge gas.

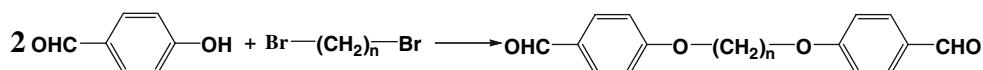
X-ray diffraction(XRD) measurements were performed on powder samples using D/MAX-III C X-ray Diffractometer. The radiation source used Cu K α radiation with a wavelength of 0.154 nm. The voltage was set to be 40 kV and the current 40 mA. Samples were placed on a sample holder and scanned from 3 to 50° in 2θ at a speed of 5°/min.

Syntheses of dialdehydes of monomer

Dialdehydes were synthesized according to the reactions presented in Scheme 1.

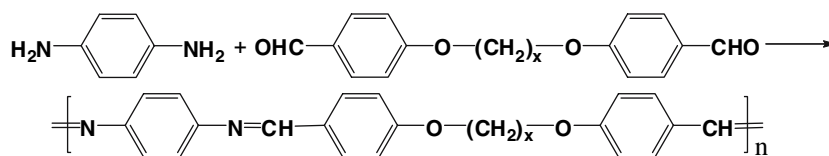
4,4'-Diformyl- α , ω -diphenoxyalkane were prepared as follows [22]: Anhydrous sodium carbonate (0.25 mol) and the appropriate dibromoalkane (0.1 mol) dissolved in DMF (25 ml) were added into a 250 ml three-necked flask equipped with a condenser and magnetic stir bar. Then, a solution of 4-hydroxybenzaldehyde (0.2 mol) in DMF (25 ml) were also added to the reaction flask. The mixture was stirring and then refluxed at 120 °C for 4 h. After cooling, the product was poured into cold water (about 500 ml) and allowed to stand overnight in a refrigerator. The precipitate was filtered off, washed with water, dried under vacuum and recrystallized from ethanol.

4, 4'-(propane-1, 3-diylbis(oxy))dibenzaldehyde($n = 2$). Yield: 55%. M.p.: 110–112 °C. ^1H NMR (DMSO- d_6) (δ , ppm): 9.79(s, 1H, -CHO), 7.19–7.91(s, 8H, aromatic protons), 4.50(t, 4H, -O-CH $_2$ -).



Scheme 1 Synthesis of the aliphatic-aromatic dialdehydes M $_1$: $n = 2$ M $_2$: $n = 4$

Scheme 2 Synthesis of poly(Schiff base)s P₁: $x = 2$ P₂: $x = 4$



4, 4'-(butane-1, 4-diylbis(oxy))dibenzaldehyde ($n = 4$): Yield: 60%. M.p.: 102–104 °C. ¹HNMR (DMSO-*d*₆) (δ , ppm): 9.88(s, 1H, -CHO), 7.13–7.89(s, 8H, aromatic protons), 4.18(t, 4H, -O-CH₂-), 1.93(m, 4H, -CH₂-).

Synthesis of polymers of Schiff base

The synthesis of polymers and the structure of the poly(Schiff base)s are given in Scheme 2.

1, 4-phenylenediamine (1 mmol) dissolved in absolute ethanol (20 ml) were added into a 250 ml three-necked flask equipped with a condenser and a magnetic stir bar. Then, the dialdehydes (1 mmol) was added to this solution. The mixture was stirred 6 h at reflux under dry nitrogen. The polymer precipitate was isolated by filtration, and then washed with hot methanol, hot acetone and then dried in vacuum.

Synthesis of inclusion complexes

A small amount of NMP solution of Poly(Schiff base)s (0.05 wt%) were mixed with a certain amount of saturated aqueous solution of β -CD with the ratio of 1:24 at 70 °C, and the mixture was stirred continuously for 9 h at 70 °C, followed by standing overnight at room temperature. The white crystalline inclusion complex in a form of precipitate was filtered and dried in vacuum at 60 °C for at least 24 h.

Results and discussion

In this study, we used the method that the inclusion complexes were formed with the dissociated monomers (amines and aldehydes) in the inclusion complexes in the preliminary study. But this method need a long reactive time and the products could not use the common solvent to purify because of the poor solubility of the products. So the polymer-CD-ICs were synthesized through threading of β -cyclodextrin onto the polymers.

Because neither polymer is soluble in common organic solvents, their molecular weights could not be obtained. We did not put up viscosity measurements because the solubilization of poly(Schiff base)s in sulphuric acid leads to the modification of their structure (scission of

azomethine linkage). This is clearly indicated by the IR spectra recorded for 1,3,4-oxadiazole containing polyazomethine before and after viscosity measurements (polymers prepared by Yasuo Saegusa et al.) [23].

There are visible changes in the spectra of two polymers in comparison with the spectra of the corresponding monomers, respectively. It is observed from Fig. 1 and 2 that some signals disappeared or weakened in the amplitude. Two monomers present characteristic peaks for terminal aldehyde group at 1683 cm⁻¹, however, in the IR spectra of polymers they are diminished and the strong signal at 1602 cm⁻¹ clearly indicates the -CH=N- link formed during the polymerization process. 1246(P₁), 1253(P₂) cm⁻¹ and 1456(P₁), 1427(P₂) cm⁻¹ were for ether absorption band of ph-O-C and C-H in the aromatic ring, respectively. The absorption peak at 1510 cm⁻¹ was denoted for stretching vibration of C-C in the aromatic ring.

It has been demonstrated that FTIR is a very useful tool to prove that there are supramolecular interactions between host and guest in inclusion complexes. Figure 3 and 4 present the FTIR spectra of β -CD, polymer and polymer-CD-ICs in the region from 4,000 cm⁻¹ to 400 cm⁻¹.

The pure β -CD spectrum shows the characteristic peaks at 3,391 cm⁻¹ was assigned to symmetric and

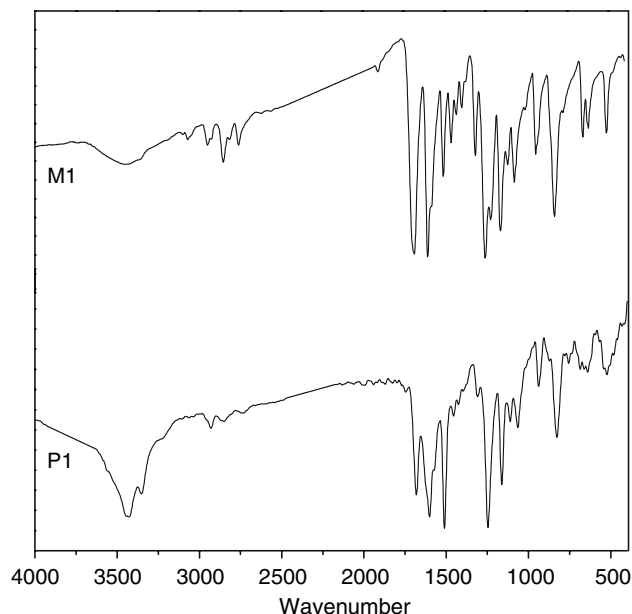


Fig. 1 IR spectra of P₁ and M₁

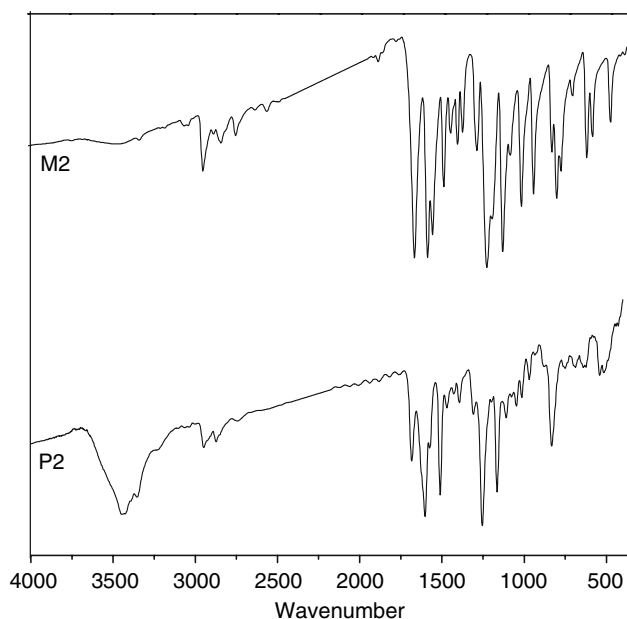


Fig. 2 IR spectra of P₂ and M₂

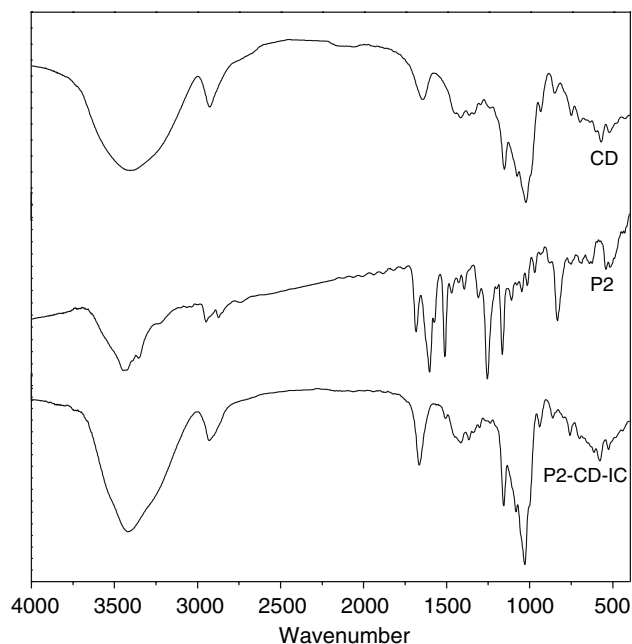


Fig. 4 IR spectra of β-CD, P₂ and P₂-CD-IC

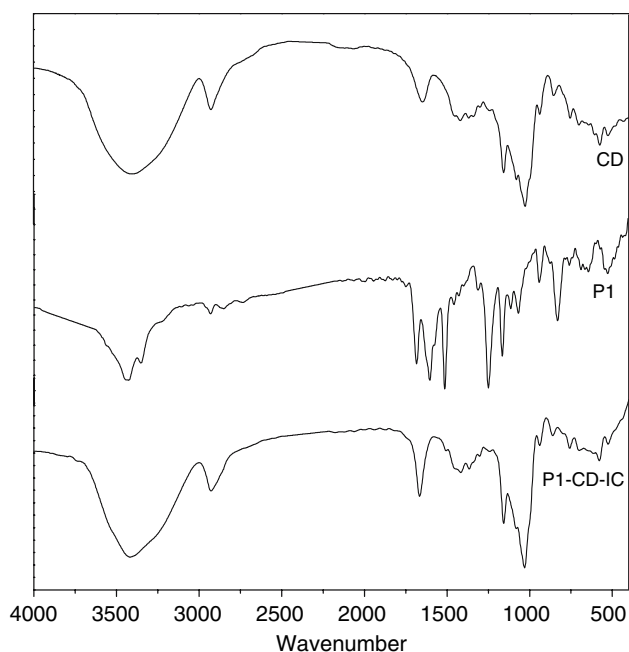


Fig. 3 IR spectra of β-CD, P₁ and P₁-CD-IC

antisymmetric O–H stretching modes. It shifted to higher frequency at $3,418\text{ cm}^{-1}$ when β-CD formed the inclusion complexes with poly(Schiff base)s, this may be caused by the non-covalent interaction between β-CD and the polymer backbone. In the polymers' spectrum, the intense peak at $1,602\text{ cm}^{-1}$ attributed to the $-\text{CH}=\text{N}-$ stretching vibration diminished after formation of IC. This may be proved that there was interaction between β-CD and polymer. The peak at $1,029\text{ cm}^{-1}$ was greatly enhanced,

which showed that bend vibration of O–H in the molecule of β-CD was increased. The peak at $1,157\text{ cm}^{-1}$ was also greatly enhanced, which showed that the C–C bond was stretched in the ring of β-CD. These results might be caused by the effect between poly(Schiff base)s and β-CD and indicated the formation of the inclusion complexes between poly (Schiff base)s and β-CD.

¹H-NMR spectroscopy is found to be the most effective tool for the study of inclusion complexes formation between cyclodextrin and guest molecules [24]. To attribute to the poor solubility of the poly(Schiff base)s in common organic solvents, their ¹H-NMR spectra could not be properly recorded. There were only obtained and compared the β-CD and inclusion complexes' ¹H-NMR spectra.

Usually slight changes in the chemical shifts of β-CD can be observed due to the change of chemical environments as a consequence of host-guest interaction or inclusion complex. The resonances of β-CD protons located in the interior of the cavity (H-3 and H-5) show remarkable chemical shifts upon inclusion of the guest molecule. A small shift is observed for the resonances H-1, H-2 and H-4, which are in the exterior of the cavity. The H-6 protons are at the smaller rim of the cavity and in most cases show a chemical shift slightly larger than those of the exterior protons (H-1, H-2 and H-4).

¹H-NMR spectra of pure β-CD and polymer-CD-ICs are given in Fig 5. Furthermore, the change in chemical shifts of six protons of β-CD was showed in Table 1. It is well known that the H-3 and H-5 protons are located in the interior of the β-CD's cavity and the inclusion of polymers

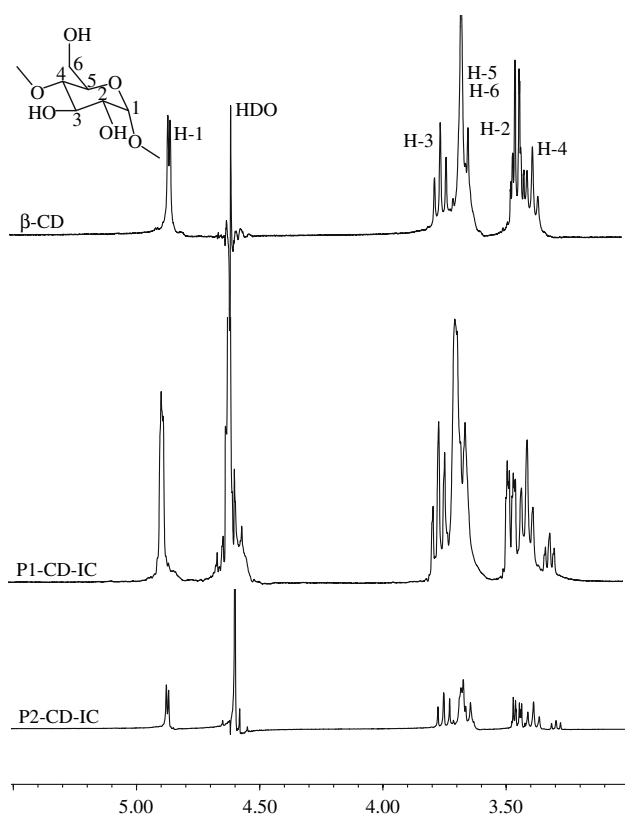


Fig. 5 $^1\text{H-NMR}$ spectra of $\beta\text{-CD}$, $\text{P}_1\text{-CD-IC}$ and $\text{P}_2\text{-CD-IC}$ in D_2O

Table 1 Variation in $^1\text{H-NMR}$ chemical shift of $\beta\text{-CD}$ and polymer- CD-IC in D_2O

	H-1	H-2	H-3	H-4	H-5	H-6
$\delta(\beta\text{-CD})(\text{ppm})$	4.853	3.411	3.740	3.386	3.627	3.655
$\delta(\text{P}_1\text{-CD-IC})(\text{ppm})$	4.869	3.412	3.772	3.389	3.674	3.681
$\Delta\delta(\text{ppm})$	0.016	0.001	0.032	0.003	0.047	0.026
$\delta(\beta\text{-CD})(\text{ppm})$	4.853	3.411	3.740	3.386	3.627	3.655
$\delta(\text{P}_2\text{-CD-IC})(\text{ppm})$	4.866	3.412	3.777	3.389	3.671	3.676
$\Delta\delta(\text{ppm})$	0.013	0.001	0.037	0.003	0.044	0.021

with $\beta\text{-CD}$ will affect the chemical shifts of these two protons. Compared with $\beta\text{-CD}$, the chemical shift of the interior protons H-3, H-5 in the inclusion complexes have shifted to higher field, which is perhaps due to the direct interaction of these protons with polymers. In contrast, the chemical shifts of outer protons H-2 and H-4 are relatively unchanged, indicating the interaction occurs inside the cavity. Therefore, this may provide the evidence for the formation of the inclusion complexes.

The thermal stabilities of $\beta\text{-CD}$, polymer and polymer- CD-IC s were investigated by thermogravimetric analysis (TGA). Figure 6 and 7 display the thermogravimetric analysis for $\beta\text{-CD}$, polymer and polymer- CD-IC s up to

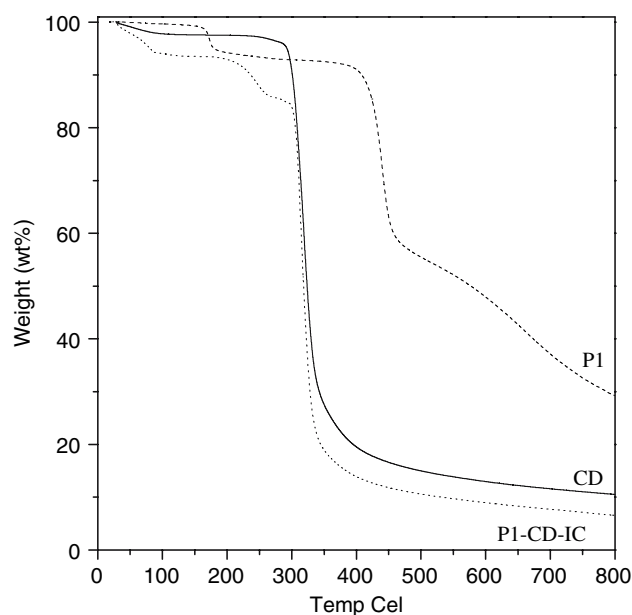


Fig. 6 The TGA trace of $\beta\text{-CD}$, P_1 and $\text{P}_1\text{-CD-IC}$

800 $^{\circ}\text{C}$, respectively. Two polymers have good thermal stability and similar behavior over the whole degradation process. The decomposition point of polymers are at 408 $^{\circ}\text{C}$, 63% (P_1) of original weight was lost at 788 $^{\circ}\text{C}$ and 52% (P_2) of original weight was lost at 792 $^{\circ}\text{C}$. This means that the small difference in spacers (only two methylene groups) has little influence on their thermal stability. They have two stage during the degradation process, based on their similar structures, due to the second stage, the $-\text{CH} = \text{N}-$ linkage is breaking.

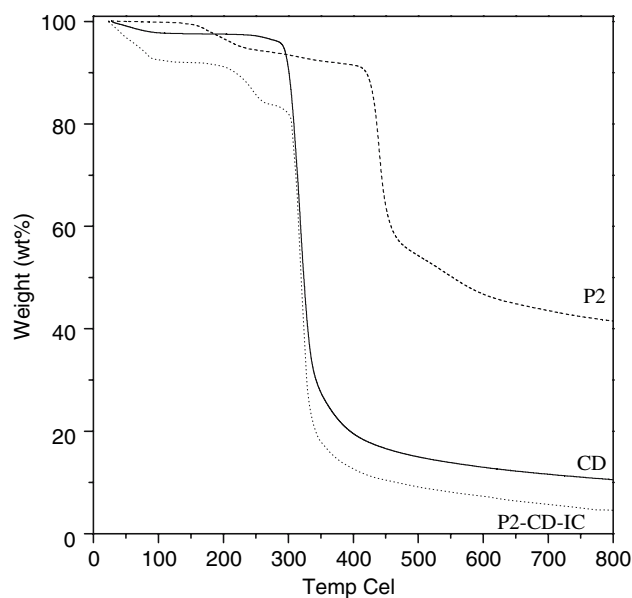


Fig. 7 The TGA trace of $\beta\text{-CD}$, P_2 and $\text{P}_2\text{-CD-IC}$

In comparison with polyamide-azomethine ethers [25] and the more pronounced rigidity of the polymers, which have the liquid crystalline properties, no liquid crystalline properties were observed in these two polymers. This phenomenon may be due to the presence of the spacers.

The polymer-CD-ICs undergo three stage thermal degradations and lose approximately 95% of their original weight at 788 °C. The first step may be due to adsorptive water or other low molecular weight substance and the third step can be mainly attributed to decomposition of polymers, and the second step mainly due to the decomposition of β -CD. Although the polymer-CD-ICs and free β -CD start to decompose at similar temperatures 275 °C, but there is no overlapping step towards slightly higher temperature for the degradation of polymer, the course of weight loss for the complexed CD is obviously slower than free β -CD. This phenomenon may imply that the polymer chains included inside the polymer CD-IC channels can improve β -CD's thermal stability, and the polymers' thermal stability is depressed result that the practical applications of this kind of polymer is accessible. The decomposition of β -CD, polymer and polymer-CD-ICs is an additional support for the successful isolation of a true inclusion compound, because it might suggest that the polymer chains are isolated from one another by β -CD molecules.

X-ray diffraction was used to determine the structure period of inclusion complex. Crystals of β -CD could be mostly categorized into two types, cage-type and channel-type [26]. In cage-type structure, β -CD molecules are arranged in a herringbone fashion, and both ends of the β -CD cavity are blocked by adjacent molecules [27]. The channel-type structure is formed by the linear stack of β -CD rings. The column-like structure can include molecules that are longer than the depth of the β -CD cavity and penetrating two or more β -CD rings. The channel-type structure is further classified into two types, the head-to-head and the head-to-tail. Only head-to-head type was in accordance with the axial structure period of β -CD with two times of the cavity height [28].

Figure 8 and 9 present the comparison of X-ray diffraction patterns observed for β -CD, polymers and polymer-CD-ICs at room temperature from $2\theta = 3$ to 50° . The most intense peak of β -CD (relative intensity $I/I_0 = 100$) was in the position of $2\theta = 12.98^\circ$ ($d = 6.81 \text{ \AA}$, the depth of the β -CD cavity), while in the position of $2\theta = 6.54^\circ$, the d value was 13.50 \AA , two times the depth of the β -CD cavity, which could be deemed to channel-type packing.

The polymer-CD-ICs showed a diffraction pattern quite different from the diffractograms of polymers and β -CD. These patterns indicate that both of these inclusion complexes are completely different from β -CD and polymers

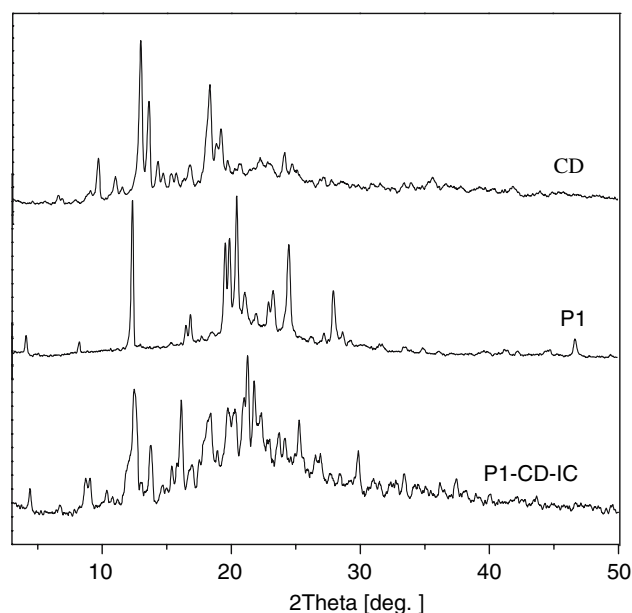


Fig. 8 XRD patterns of β -CD, P₁ and P₁-CD-IC

and showed the formation of a new crystalline phase, different from either of β -CD or polymer. The appearance of two sharp peaks in P₁-CD-IC at $2\theta = 12.44^\circ$ ($d = 7.11 \text{ \AA}$), 21.28° ($d = 4.17 \text{ \AA}$) and P₂-CD-IC at ca. $2\theta = 12.72^\circ$ ($d = 6.95 \text{ \AA}$), 21.40° ($d = 4.15 \text{ \AA}$) may be the key feature to indicate the channel-type structures of the β -CD/polymer inclusion complex [29]. We may need the further study on these polymer-CD-ICs, because of their complicated diffraction pattern, but the results here indicate that, in the polymer-CD-ICs sample, the β -CD exhibits a different packing from that in free β -CD.

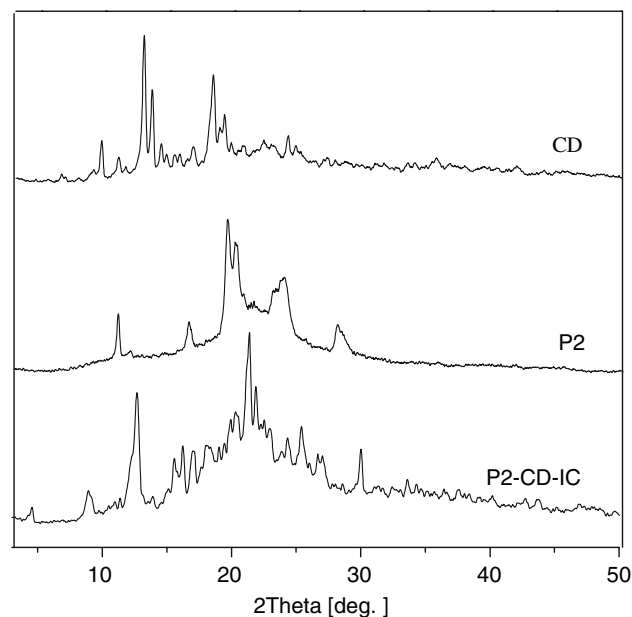


Fig. 9 XRD patterns of β -CD, P₂ and P₂-CD-IC

Conclusions

In this study, the inclusion complexes between β -cyclodextrin (β -CD) and aliphatic-aromatic poly(Schiff base)s have been successfully synthesized by solution-heating technique. The formation of polymer-CD-ICs was observed through FTIR, $^1\text{H-NMR}$, TGA and XRD. In FTIR spectra, the characteristic peaks of β -CD and the intense peak of polymers diminished after formation of inclusion complexes. From the $^1\text{H-NMR}$, the change of the protons' chemical shift may indicate the interaction of the protons with polymers. The TGA analysis suggested that the polymers' thermal stability is depressed and the practical application of polymer is accessible. The XRD patterns displayed that the strong characteristic peak for both polymer-CD-ICs may confirm their IC formation. At the same time, the active hydroxyls of polymer-CD-ICs provided the possibility of reacting to other molecules.

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