Synthesis and characterization of poly(butanediol sebacate-butanediol) terephthalate (PBSeT) reinforced by hydrogen bond containing amide group, with good mechanical properties and improved water vapor barrier

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Abstract
A PBSeT copolyester was synthesized by introducing a polar amide unit into the molecular chain, coordinating with decyl-diamine through hydrogen bonds between the amide groups and hydrogen atoms on the chains. Two-step esterification and one-step polycondensation melt polymerization were adopted. ATR-FTIR and HNMR demonstrated the successful introduction of the amide unit. The effect of adding an amide unit on many properties was also discussed. Due to the formation of hydrogen bonds, both the force of molecular chain and the crystallinity increased by 5.91%. Consequently, yield and tear strength increased by 40.7% and 74.8%, respectively. The addition of 1,10-decyldiamine has no significant effect on the glass transition temperature and thermal stability of polyester. The introduction of an amide unit decreased free volume and increased combined crystallinity, which also increased the water vapor barrier. At the same time, PBSeT copolyester still maintained its biodegradability under hydrolysis and enzymatic hydrolysis. Additionally, the change of surface morphology upon the degradation was examined by AFM. This work discussed a synthesis method of PBSeT copolyester strengthened by the hydrogen bond. The modified material has higher crystallinity, better mechanical properties, and a better water vapor barrier while maintaining certain biodegradation ability. It can be used in disposable products, food packaging, and other fields.

Keywords 1,10-Decyldiamine · Water vapor barrier Properties · Copolyesters

1 Introduction
With the rapid development of traditional plastics, a large number of discarded plastics and their subsequent disposal have become an urgent problem to be solved [1–4]. The concept of environmental protection is constantly upgraded and optimized around the world, and the concept of sustainable development is put forward, which makes bio-based and biodegradable materials stand out among many traditional plastics [5–12]. Some aliphatic copolyesters have been widely used for their excellent biodegradability because they can be decomposed into carbon dioxide and water under the action of microorganisms. Poly(succinic acid) (PBS), poly(lactic acid) (PLA), and poly(1,6-hexanediol adipate-co-terephthalate) (PBAT) are representative products of aliphatic biodegradable materials, which have been recognized by the market and enterprises in many fields such as food packaging and disposable products [13–15]. The above products still have
problems that need to be solved. The raw materials are still from the petrochemical industry, and the performance is insufficient [16–20].

Since the advent of aliphatic–aromatic biodegradable copolyesters, researchers have been troubled by problems such as low crystallinity, poor mechanical properties, and poor gas barrier [21–23]. In order to make aliphatic and aromatic biodegradable copolyesters achieve the performance of traditional plastics, the introduction of an amide unit into the original molecular chain is a simple and effective way to improve the performance. Physical blending is usually the most direct way to introduce amide units into polyester materials [24–29]. This will make polyester material with higher crystallinity and improved mechanical and barrier properties. Yan et al. mixed bio-based polyamide 56 (PA56) with polyethylene terephthalate (PET) as a sustainable product candidate to replace commercial PA6 and PA66 and obtained higher crystallinity and better compatibility [30]. Hu et al. improved the gas barrier of PET by blending it with aromatic polyamides [31]. This method also has a disadvantage in that the compatibility of the two phases of the blend is not good enough to lead to performance degradation [32]. In such cases, compatibilizers must be used for improvement [33]. Therefore, it may be better to introduce amide groups, similar to polyester amide structures, into the polymer synthesis process by chemical synthesis [34]. In this way, hydrogen bonds can be introduced into the original polymer molecular chain, resulting in a physically crosslinked structure, improving performance [35]. Li et al. investigated the physical crosslinking of poly (butylene sebacate-butylene terephthalate) (PBSeT) with different glycerol contents by introducing a hydrogen bond structure to obtain better mechanical properties [36]. Gao et al. obtained PET film with better mechanical properties and gas barrier by introducing hydrogen bond in PET molecular chain copolymerization of 1.12-dodecanediamine [37]. Inspired by the above development, 1,10-decylidiamine was selected as the bio-based monomer to explore the effect of a small amount of addition on the performance of aliphatic–aromatic copolyesters in order to prevent it from no longer having biodegradability.

In this study, aliphatic–aromatic copolyester: poly (butylene sebacate-butylene terephthalate) (PBSeT) is a modified product of PBAT, synthesized with sebacic acid, terephthalic acid, butylene glycol, and a small amount of sebacic acid as the monomer. Sebacic acid and sebacidiamine, as bio-based chemical materials extracted from castor oil, have become important monomers to replace petrochemical raw materials. Both are made from castor oil through a series of processes such as hydrolysis, pyrolysis, and acidification. Through the reaction of the amine group and a carboxyl group, the amide bond is formed. Then the intermolecular hydrogen bond is formed to improve the water vapor barrier performance and mechanical properties of PBSeT copolyester while ensuring its biodegradability. This work mainly solves the problem of insufficient water vapor barrier of biodegradable copolyester PBSeT. When it is used as a mulch film, water vapor is lost and crops cannot grow normally. It can be applied to food packaging and disposable plastic supplies. Decylenediamine was introduced into biodegradable copolyesters to improve various properties while retaining certain biodegradability. It provides a way to solve the application problem of biodegradable copolyesters.

2 Experimental

2.1 Materials

Sebacic acid (SeA) was kindly provided by Hengshui Jinhua Chemical Industry Co., Ltd. Terephthalic acid (TPA), 1,4-butanediol (BDO), 1,10-decylidiamine (DA) (Zeyuan Biotechnology Co., Ltd. (Shanxi, China)), tetra butyl titanate (TBOT, Wuxi Yatai United Chemical Co., LTD.), glycerol (Aladdin Reagent Co., Ltd. (Shanghai, China)) chloroform (Tianjin Shentai Chemical Reagent Co., LTD.), phenol (Guangzhou Chemical Reagent Factory), tetrachloroethane (Shanghai Chemical Industry Park), and methanol (Tianjin Hengxing Chemical Reagent Manufacturing Co. LTD) were all used as received.

2.2 Synthesis of PBSeT containing amide groups

Hydrogen-bonded PBSeT copolyester was prepared by adding 1,10-diaminododecane as the fourth monomer through two-step esterification and one-step polycondensation (Scheme 1). 1,10-Diaminododecane added amount of terephthalic acid substances accounted for 0–1% of the amount. The corresponding reaction parameters are given in Table 1. In the first esterification stage, butanediol (BDO) (3.5 mol), terephthalic acid (TPA), glycerin (0.1% acid content), and 1,10-diaminododecane were reacted for 1.5 h under mechanical agitation at 180 °C in a 5-L reactor. The effect of adding glycerol is to shorten the reaction time and accelerate the reaction process. Therefore, the performance improvement of the modified product compared with the control group naturally comes from the introduction of the amide bond. In the second esterification stage, the reaction temperature was raised to 220 °C then sebacic acid, catalyst TBOT (0.25% acid content), and remaining BDO (1.5 mol) were added simultaneously. The reaction lasted for 4 h. The amount of terephthalic acid and sebacic acid was 4.55 mol and 2.45 mol, respectively, with a ratio of 6.5:3.5. When the discharge water content reaches the theoretical value, TBOT (0.05% acid content) is added to
the reaction kettle and vacuumized to enter the stage of polycondensation. During the polycondensation stage, the temperature was controlled at 250 °C for 4.5 h. When the torque of the control box of the reaction kettle remained unchanged, the reaction products were obtained at the end of the reaction. Samples were dissolved in chloroform and precipitated in supercooled methanol. The final product was obtained after drying at a temperature of 40 °C for 24 h.

The unmodified PBSeT was named C.S. The modified samples with different addition amounts of decanediamine were named DA-0.2, DA-0.4, DA-0.6, DA-0.8, and DA-1.0.

### 2.3 Characterizations

ATR-FTIR spectra of the copolyesters were recorded with a Nicolet iS10 spectroscopy (Thermo Fisher Scientific, USA) equipped with a ZnSe crystal ATR accessory. The scan range

![Scheme 1](image)

**Scheme 1** Schematic diagram of the synthesis of PBSeT-DA

| Sample  | Φ_{DA} (mol) | T_{es}/t_{es} | T_{mp}/t_{mp} | [η]Ω (dL/g) | GPC |
|---------|---------------|---------------|---------------|--------------|-----|
|         |               |               |               |              | Mn (g/mol) | Mw (g/mol) | PDI  |
| PBSeT   | 0             | 220/3.5       | 240/6         | 1.09         | 50,379     | 64,989     | 1.29 |
| DA-0.2% | 0.0091        | 180/1.5 + 220/2.5 | 240/5.5      | 1.05         | 53,212     | 65,994     | 1.24 |
| DA-0.4% | 0.0182        | 180/1.5 + 220/2.5 | 240/5         | 1.11         | 53,379     | 61,920     | 1.16 |
| DA-0.6% | 0.0273        | 180/1.5 + 220/2.5 | 240/5         | 1.25         | 60,132     | 75,165     | 1.25 |
| DA-0.8% | 0.0364        | 180/1.5 + 220/2.5 | 240/5         | 1.20         | 52,219     | 61,618     | 1.18 |
| DA-1.0% | 0.0455        | 180/1.5 + 220/2.5 | 240/4.5       | 1.13         | 55,617     | 61,179     | 1.10 |

*a* (1) Esterification: TBT, 0.6 mol% based on diacid was used as catalyst; diol/diacid molar ratio 1.2.

*b* (2) Polycondensation: additional 0.3 mol% TBT was added

*c* The moles of 1,10-decyldiamine

*d* Esterification temperature and time

*e* Melt polycondensation temperature and time

*f* Intrinsic viscosity was measured at 25 °C using a mixture of phenol-tetrachloroethane with a mass ratio of 1:1 as solvent

![Table 1](image)

**Table 1** Synthetic conditions and results of the eight poly(butylene sebacate-co-terephthalate)s
was from 500 to 4000 cm\(^{-1}\), and each sample was scanned 32 times. Disk specimens were prepared using a precision thermostat table and a hot-press molding at 180 °C.

\(^1\)H NMR spectra were recorded with a Bruker AVANCE-3 HD spectroscopy (600 M). Deuterated chloroform was used as solvent and tetramethylsilane as the internal reference. After the NMR tube was cleaned by ultrasonic vibration, deuterium chloroform was used as a solvent to dissolve the sample, and tetramethylsilane was used as the internal reference for testing.

Polydispersity (PDI), the number-average molecular weight (\(M_n\)), and weight-average molecular weight (\(M_w\)) were determined at 35 °C with a gel permeation chromatography (GPC, Waters Co., USA) equipped with a differential refractive index detector. Tetrahydrofuran was used as an eluent at a flow rate of 1.0 mL/min.

The intrinsic viscosity [\(\eta\)] of all samples dissolved in the phenol /1,1,2,2-tetrachloroethane mixture was measured using a Ubbelohde viscometer at 25 °C. The inner diameter of the capillary tube of the Ubbelohde viscometer is 0.88 mm. The mass ratio of phenol to 1,1,2,2-tetrachloroethane is 1:1.

Thermal transition behaviors were recorded with differential scanning calorimetry (DSC-1, METTLER TOLEDO) under a nitrogen flow condition. Approximately 7–8 mg of the sample was tested under a standard heat-cool-heat cycle from −60 to 200 °C. Both heating and cooling rates were 10 °C/min. The temperature was kept at 200 °C for 5 min before cooling and the thermal history was eliminated.

X-ray diffraction (XRD) pattern was recorded using a RIGAKU X-ray diffraction system (D/max-Rb) with Cu radiation (1.54 Å), working at 40 K.V. and 100 mA. The samples were scanned from 20 = 5° to 20 = 50° with a step size of 0.01° and an acquisition time of 30 s per step.

Thermogravimetric analysis (TGA) measurements were carried out under an argon atmosphere by a thermal analyzer (TGA, TG290, NETZSCH) from 10 to 600 °C at a 10 °C/min heating rate.

Tensile properties were measured with a CMT6104 universal testing machine at a tensile speed of 50 mm/min. Dumbbell-shaped tensile specimens were prepared by injection molding. All the specimens were kept in a standard atmosphere of 25 °C for at least 24 h before testing. At least five specimens were tested for each sample, and all the reported properties were presented with average values of all the trials.

According to GB 1037–1988, the cup method is used to measure the sheet metal’s water vapor transmittance (WVTR). Dry CaCl\(_2\), a hygroscopic cup, and the sample film are then covered and sealed with sealing wax. The sealing wax formula is a mixture of 85% paraffin wax and 15% beeswax. Place the permeable cup in an incubator with a constant temperature (38 °C) and humidity (90 RH%). Record the total weight of the test cup over time. Due to the strong hygroscopicity of dried CaCl\(_2\), the relative humidity inside the cup is close to zero, so the added weight is close to the weight of the water passing through the sample. Due to the addition of anhydrous calcium chloride as water absorbent and desiccant inside the sample, the relative humidity can be approximately regarded as 0%. The whole moisture permeable cup is placed in the biochemical incubator to ensure the stable temperature and humidity conditions of the external environment. This controls the relative humidity on both sides. Water vapor permeability PWV was calculated with Eq. (1), where \(d\) is the thickness of the film, WVT is water vapor transmission, \(P_0\) is the saturated water vapor pressure (6630 Pa at 38 °C), and RH\(_1\) and RH\(_2\) are the relative humidities on both sides of the film.

\[
P_{\text{WV}} = \frac{\text{WVT} \times d \times (\text{RH}_1 - \text{RH}_2)}{\text{d} \times \text{p}_0} \tag{1}
\]

Co-polyester in the form of films with a 1 cm\(^2\) area and 0.3 mm in thickness were prepared by a hot-press molding at 180 °C with a precision thermostat. The enzymatic degradation test was conducted by placing the sample in a vial containing 0.8 mg/mL lipase phosphate buffer (pH 7.4) at 37 °C. The hydrolysis degradation experiment was conducted by placing the sample in a phosphoric acid medium with pH=4.4 and 9.6 at 37 °C. The media were replaced every 7 days. The films were removed from the vial every 7 days, washed with distilled water, dried under a vacuum, and weighted until they reached a constant weight. The degree of biodegradation was estimated from the weight loss.

Using the sitting drop method of automatic microscopic,contact angle tester (OCA20 from Dataphysics, Germany). A micro sampler was used to add 4 µL of deionized water to the surface of the plastic sample, and the measurement time was not more than 30 s. The average value of the five measurement results was taken as the contact angle value of the plastic sample.

The change in the surface morphology upon degradation was evaluated by scanning an atomic force microscope (AFM, Cypher E.S. from AsylumResearch, USA). The sample is a degraded copolyester sample with an area of 1 cm\(^2\) and 0.3 mm in thickness. All AMF data are processed by Gwyddion software, in which all sample roughness curves are uniformly obtained on the diagonal of the sample.

\section{3 Results and discussion}

\subsection{3.1 Synthesis and molecular weight characterization}

The intrinsic viscosity of copolyester was measured by phenol/tetrachloroethane mixture solution (1/1 W/W) and the
number mean molecular weight and PDI of copolyester are measured by GPC. The reaction conditions and test results of copolyesters are shown in Table 1.

The esterification reaction is divided into two sections for reaction. In the first section of esterification, only add terephthalic acid, a monomer, to 1,10-decyldiamine and terephthalic acid to react fully. At the same time, the sum of the first and second esterification times is also greater than the time of non-staged esterification to ensure that the esterification reaction is fully cut and complete. The intrinsic viscosity of all polymers is greater than 1, and the mean molecular weight of all copolyesters is greater than 50,000. Compared with C.S., the molecular weight of PBSeT modified has been improved to different degrees, and the trend is similar to the characteristic viscosity. In conclusion, copolyesters have a high molecular weight.

3.2 Structural characterization and hydrogen bond formation in PBSeT-Da

$^{1}$H NMR spectra of copolyester samples obtained by copolymerization with decyldiamine as the fourth monomer are shown in Fig. 1. The test results of characteristic peaks of hydrogen atoms in the structure are consistent with the expected results. Characteristic absorption peaks a and b are the different absorption peaks of hydrogen splitting in the methylene group near and away from the ester group in butanediol linked to different units, respectively. Both a and b split into four different absorption peaks, $a_1$-$a_4$ and $b_1$-$b_4$. Compared with the $^{1}$H NMR spectra of unmodified PBSeT copolyester, two new characteristic peaks, g and e, appeared in PBSET-DA. The characteristic peak of g at $\delta = 3.75$ is the characteristic absorption peak of the hydrogen atom in the methylene group adjacent to the amide bond in 1,10-diaminododecane. The characteristic peak of E at $\delta = 7.90$ is the characteristic absorption peak of the hydrogen atom on the amide bond. The e and d characteristic peaks in the DA-1% curve were analyzed by MestreNova software. It is found that the area ratio of absorption peak d and absorption peak e of the hydrogen atom in benzene ring is 1:0.0096. This indicates that the amide group of 1,10-Diaminododecane, which accounts for 1% of TPA, was successfully incorporated into the main chain of PBSeT copolyester.

The structure of untreated PBSeT and PBSeT with different proportions of decyldiamine were characterized by ATR-FTIR spectroscopy. The result is shown in Fig. 2. All samples had C–O–C stretching vibration and C-O contraction vibration at 1104 cm$^{-1}$ and 1216 cm$^{-1}$, respectively. The strong signal at 2854 cm$^{-1}$ and 2924 cm$^{-1}$ is a distinct aliphatic band, both of which testify to the presence of ester groups. Unlike untreated PBSeT, new amide-I and amide-II absorption bands were observed in THE ATR-FTIR spectra of DA (Fig. 2C). The the ATR-FTIR spectrum results

![Fig. 1 $^{1}$H NMR spectra of the PBSeT and PBSeT-DA](image)
consistent with the $^1$HNMR test results, which further proved that the fourth monomer 1,10-diaminodecane was introduced into the main chain. The addition of glycerol resulted in a wide hydroxyl absorption band at 3400 cm$^{-1}$ in all samples (Fig. 2B). It is because the secondary hydroxyl group in glycerol is not easy to participate in the reaction. It is easier to form hydrogen bonds with the hydrogen atom in the amide group and produce physical crosslinking under this action. The formation of a physical crosslinking network is proved.

3.3 Thermal transition

In order to verify whether the introduction of amide groups and the formation of hydrogen bonds influence the thermal

![ATR-FTIR spectra](image)

**Fig. 2** ATR-FTIR spectra of the PBSeT(CS) and PBSeT-DA in the range of 3600–300 cm$^{-1}$ (A), 3600–3050 cm$^{-1}$ (B), and 1750–1500 cm$^{-1}$ (C).

![DSC thermograms](image)

**Fig. 3** DSC thermograms of PBSeT(CS) and PBSeT-DA: A cooling scan at $-10$ °C/min, B 2nd heating scan at 10 °C/min.
properties of PBSeT copolyesters, DSC and T.G. tests were carried out on the samples. The results of DSC testing the melting and cooling behavior of copolyesters are shown in Fig. 3, and the specific values are shown in Table 2. At the cooling and heating rate of 10 °C/min, all samples have crystallization and melting peaks, and all have a single Tg. This shows that the different components of polyester chain segment have good compatibility. During cooling scanning, with the increase of DA content, the maximum temperature Tc corresponding to the exothermic peak produced by crystallization increases continuously, while ΔHc also shows a rising trend. In the crystallization process, the large molecular chain where the benzene ring resides is embedded in the lattice, and multiple chain segments cooperate to complete the crystallization process. The crystallinity of co-polymers generally showed an increasing trend, as shown in Fig. 4. The crystallinity of Xc is 14.97~16.28%. When the content of sebacediamine was 6%, the crystallinity of PBSeT-Da copolyester reached 16.28%. The possible reason is that the existence of a hydrogen bond promotes the synergy between molecular chains and thus improves the crystallization capacity [38].

The melting peak and glass transition temperature appeared in the second heating process of copolyester. The glass transition temperature Tg changed little, while the melting temperature first decreased and then increased. It is worth noting that the cold crystallization temperature is constantly decreasing, and the temperature of combined melting crystallization is constantly increasing, indicating that the crystallization speed of PBSeT copolyester is constantly accelerating with the introduction of DA.

In order to further study the influence of the introduction of DA on the crystallization behavior of PBSeT, the samples were tested by XRD. XRD patterns are shown in Fig. 5. It can be clearly seen from the XRD pattern that PBSeT and PBSeT-Da have a similar crystal structure and belong to semi-crystalline polymers with wide diffraction peaks. The XRD patterns of PBSeT were peak-fitting. At 20, 16.3°, 17.5°, 20.5°, 23.1°, and 24.9°, the characteristic diffraction peaks corresponding to the crystal planes were (011), (010), (101), (100), and (111), respectively. The crystallities of copolyesters grow on different crystal planes, and the peak strength of (100) crystal plane reaches the maximum, which is similar to the crystallization law of PET, proving that the crystallization capacity of PBSeT copolyesters is mainly provided by B.T. unit [39, 40]. The peak area of the spectrum is also consistent with the relative crystallinity obtained by DSC. The crystal plane position of PBSeT did not change significantly, indicating that the addition of decyldiamine did not change the crystal structure of PBSeT.

| Sample  | Cooling scan | Second heating scan | Xc (%) |
|---------|--------------|---------------------|--------|
|         | Tc           | ΔHc                | Tg     | Tm     |        |
| CS      | 141.64       | 25.14              | −25.33 | 172.33 | 15.38  |
| DA-0.2% | 143.41       | 24.20              | −29    | 169    | 15.28  |
| DA-0.4% | 142.23       | 29.02              | −25.67 | 170    | 16.09  |
| DA-0.6% | 143.3        | 37.94              | −27.67 | 169.33 | 16.29  |
| DA-0.8% | 143.74       | 23.23              | −29.67 | 171    | 14.97  |
| DA-1.0% | 139.9        | 28.58              | −31    | 169.67 | 15.45  |

Fig. 4 A Variation of Tm, Tc, and Tg. B Crystallinity of PBSeT(C.S.) and PBSeT-DA tested by DSC
Thermal stability

In order to further explore the introduction of amide group and hydrogen bond formation on the thermal stability of PBSeT copolyester, TGA tests were performed on all samples from room temperature to 600 °C in an N₂ atmosphere. The results are shown in Fig. 6. The decomposition temperature at 5% weight loss, maximum decomposition rate temperature, and residual weight at 600 °C are shown in Table 3.

All samples were in a stable state before 250 °C, with a weight loss rate of less than 1% and no decomposition. It is concluded that the introduction of sebacediamine does not seriously affect the thermal stability of copolyester and can meet the requirements of daily use. The main degradation temperatures of all samples were between 350 °C and 430 °C. The maximum decomposition rate (T_{dMax}) of the polymer occurs in the narrow temperature range of 404 ~ 407 °C. For the temperature at 5% weight loss, the temperature first increases and then decreases, and the temperature decreases to 368 °C at 0.8% DA. The main reason may be that with the addition of decylenediamine, the amount of methylene in the long chain of the molecular chain increases, which makes the polymer easy to decompose at high temperature, while the previous increase in decomposition temperature is due to the physical interaction in the hydrogen bond, which

![Fig. 5 XRD patterns of the indicated PBSeT and PBSeT-DA](image)

![Fig. 6 Thermogravimetric and differential thermogravimetric curves of the indicated PBSeT(CS) and PBSeT-DA (10 °C/min, Ar)](image)
improves the anti-decomposition ability of the molecular chain at high temperature. The residual coke content of PBSeT-Da copolymer was also higher than PBSeT in the test temperature range. PBSeT-DA can be safely treated without thermal degradation at temperatures 50 to 60 °C above its corresponding \( T_m \).

### Table 3 Characteristic decomposition temperatures of PBSeT(C.S.) and PBSeT-DA copolyesters

| Sample   | \( T_{d,5} \) (°C) | \( T_{d,\text{max}} \) (°C) | Residue at 600 °C (%) |
|----------|---------------------|-----------------------------|------------------------|
| CS       | 368.67              | 404                         | 2.56                   |
| DA-0.2%  | 369.33              | 406.67                      | 3.36                   |
| DA-0.4%  | 371.33              | 406                         | 6.30                   |
| DA-0.6%  | 368.66              | 404                         | 5.97                   |
| DA-0.8%  | 370                 | 403.3                       | 5.62                   |
| DA-1.0%  | 368                 | 403.3                       | 4.83                   |

### Table 4 Mechanical properties of PBSeT(CS) and PBSeT-DA

| Sample   | \( E \) (MPa) | \( \delta_b \) (MPa) | \( \delta_y \) (MPa) | \( \delta_T \) (KN/M) |
|----------|---------------|----------------------|----------------------|-----------------------|
| CS       | 45.38 ± 2.03  | 15.66 ± 0.22         | 15.66 ± 0.22         | 79.87 ± 4.73          |
| DA-0.2%  | 42.09 ± 1.75  | 20.22 ± 0.56         | 16.55 ± 0.31         | 72.85 ± 6.87          |
| DA-0.4%  | 53.77 ± 1.32  | 20.71 ± 0.47         | 17.38 ± 0.21         | 86.79 ± 8.53          |
| DA-0.6%  | 61.85 ± 0.81  | 19.43 ± 0.26         | 19.43 ± 0.26         | 100.69 ± 2.23         |
| DA-0.8%  | 68.94 ± 0.64  | 20.23 ± 0.33         | 20.23 ± 0.33         | 125.63 ± 4.35         |
| DA-1.0%  | 79.05 ± 0.91  | 22.03 ± 0.41         | 22.03 ± 0.41         | 139.61 ± 8.14         |

### 3.5 Mechanical properties

The mechanical properties of PBSeT and PBSeT-DA were characterized by a universal drawing machine. The obtained stress–strain curve and tear test results are shown in Fig. 7. The specific values are shown in Table 4, including Young’s modulus, tensile strength, yield strength, tear strength, and elongation at break.

![Fig. 7 A, B Stress–strain curves of PBSeT(CS) and PBSeT-DA. C Tear strength of PBSeT(CS) and PBSeT. D Comprehensive mechanical property curve](https://example.com/fig7.png)
Unmodified PBSeT exhibits typical mechanical properties of soft and tough semi-crystalline flexible polymers. The yield strength and elongation at the break before fracture are 15.66 MPa and 714.55%, respectively. When the amount of DA is less than or equal to 0.4%, the stress–strain curve is still soft and tough, but the elongation at break decreases to 63.56%, and the yield strength and tensile strength increase to 17.38 MPa and 20.71 MPa, respectively, which are 10.98% and 32.24% higher than those of unmodified PBSeT. It is a material that is going to be strong and tough. When the amount of DA reaches 6%, the elongation at the break of the sample plummets to 92.22%, and the yield strength comes 19.43 MPa. When the amount of DA reached 1%, the elongation at break decreased to the lowest 38.30%, and the yield strength and tensile strength reached the maximum 22.03 MPa, which increased by 40.68% year-on-year. It is noteworthy that the tensile strength of some samples decreased, presumably because the introduction of decylenediamine brought a more long carbon chain structure and reduced molecular chain rigidity. The improvement of PBSeT-Da can be attributed to the introduction of amide groups and the formation of hydrogen bonds, resulting in stronger interactions than the unmodified PBSeT molecular chain. However, there are many factors affecting the mechanical properties, such as the increase of crystallinity and molecular weight will improve the mechanical properties.

In the tear test of the sample, the tear strength of unmodified PBSeT is 79.88 kN/m, which is far from reaching the standard of industrial production compared with 200 kN/m of P.E. However, with the introduction of the amide group, the tear strength of samples increased to 86.80–139.61 kN/m. The maximum tear strength reaches 70% of P.E. tear strength, which can meet the standard of normal production and use. Such tearing strength should be the result of the interaction of hydrogen bond to molecular chain and the interaction of long flexible molecular chain.

### 3.6 Hydrophilicity and water vapor barrier properties

To verify the hydrophilic change of PBSeT copolyester, the water contact angle was measured at 20 °C. The result is shown in Fig. 8. Polyester materials because there are polar ester groups in the large molecular chain segment, so most polyester materials have certain hydrophilicity. That is, the water contact angle is about 80°. Unmodified PBSeT copolyesters have a water contact angle of 80.97°, which is a slightly hydrophilic material. The water contact angle decreased significantly with the addition of decylenediamine. When the amount of decylenediamine was 1%, the water contact angle was 62.67°, which decreased by 18.29°. The hydrophilicity of copolyesters was improved obviously. The obvious reason is the introduction of a strongly polar amide group into the PBSeT macro molecule chain, which also proves the successful introduction of the amide group.

The water vapor barrier of polymer samples was tested at 37 °C and 90% relative humidity by cup method. The experimental results are shown in Table 5. The barrier improvement factor (BIF) was defined as C.S./PBS-DA for PBSeT samples. The improvement in air resistance is measured by the size of the BIF. It is not difficult to see from Table 5 that, with the introduction of decyldiamine, the water vapor barrier of different proportions of modified PBSeT co-polymers is improved in different degrees. When adding 0.6% sebacediamine, the water vapor barrier ability reached the maximum. At the addition of 0.2%, the water vapor barrier decreased. The main reason is that at this ratio, the reduced crystallinity

**Table 5 Water vapor barrier Properties of PBSeT(C.S.) and PBSeT-DA**

| Sample | P_w | BIF |
|--------|-----|-----|
| CS     | 4.70*10^-14 | 1   |
| DA-0.2%| 4.72*10^-14 | 0.996 |
| DA-0.4%| 4.06*10^-14 | 1.158 |
| DA-0.6%| 3.53*10^-14 | 1.331 |
| DA-0.8%| 3.96*10^-14 | 1.187 |
| DA-1.0%| 3.84*10^-14 | 1.224 |

a Water vapor permeation: dish method, 38 °C, RH=90%
b Water vapor permeability coefficient with the unit of g·cm⁻²·s⁻¹·Pa⁻¹
c Barrier improvement factor (BIF) is defined as P_C/S/P_PBSeT-DA
d Water vapor transmission rate, at 38 °C, 90% relative humidity.
increases free volume, making it easier for water vapor molecules to pass through the macromolecular chain. For PBSeT-DA samples with lower relative crystallinity, the increase of vapor barrier is not obvious, which is also the same reason. In addition to the increase in crystallinity, it is speculated that the hydrogen bonding force between the polymer chains makes them more easily oriented. The free volume used for gas transport and chain segment movement is reduced, thus improving the water vapor barrier.

### 3.7 Biodegradation properties

In this part of the experiment, the biodegradability of PBSeT and its modified product PBSeT-Da was tested by hydrolysis and enzymatic hydrolysis. In order to simulate the degradation of different composts, the hydrolysis experiment was divided into two different pH = 4.4 and pH = 9.6. All degradation tests were incubated in phosphoric acid buffers for 28 days, with buffers replaced every 7 days. The two reagents used to prepare the phosphate culture medium are sodium dihydrogen phosphate and disodium hydrogen phosphate. The pH of pure solution of sodium dihydrogen phosphate is 4.6, and that of disodium hydrogen phosphate is 9.6. The pH value can be regulated by adjusting the content of both.

The curves of the relative weight of samples changing with time in hydrolysis experiments at pH = 4.4 and pH = 10.6 are shown in Fig. 9. In an acidic environment, the samples showed less mass loss, and only the unmodified PBSeT and DA samples with 0.2% content showed more than 2% mass loss. However, different experimental results were obtained under alkaline conditions, in which only when the addition amount of Da was more than 0.8%, the mass loss less than 2%. Compared with the acidic condition, the mass loss of each proportion was improved. This may be because the ester group is hydrolyzed into oligomers or shorter polyester segments during polyester hydrolysis [41]. The oligomer of the carboxy-terminated polyester is

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**Fig. 9** The weight loss curves of PBSeT(C.S.) and PBSeT-DA during hydrolytic degradation and enzymatic degradation
more easily dissolved and diffused into the aqueous solution under alkaline conditions. Hydrolysis is, therefore, easier under alkaline than acidic conditions [42–44]. After the addition of DA, the hydrolysis capacity of the sample decreased with the increase of the amount of DA, and almost no hydrolysis occurred in the acid and alkali environment when the amount of DA exceeded 0.8%. The main reason is that the introduction of the amide group does not lead to hydrolysis reaction, so the macromolecular chain cannot be decomposed into oligomers in the aqueous solution and then cannot be dispersed in the solution.

The enzymolysis experiment is to simulate the degradation of copolyester compost in a real soil environment. Figure 9 C shows the results of lipase degradation in phosphoric acid buffer culture. The biodegradation ability of aliphatic aromatic copolysters decreased due to the increase of benzene ring content, so the degradation ability of the copolyester sample with TPA:SeA = 6.5:3.5 was slightly lower than that of the 5:5 copolyester. The mass loss in enzymatic hydrolysis was much higher than that in hydrolysis. Lipase accelerated the hydrolysis of ester groups and led to rapid degradation of copolysters. The addition of DA leads to the degradation ability decline. The amide group’s introduction increases the molecular chain’s steric hindrance, so lipase is not easy to act on the corresponding ester group. The degree of crystallinity also affects the degradation ability of copolysters. When the crystallinity is low, lipase adhesion and enzymatic hydrolysis are more likely to occur. Enzyme-catalyzed and autocatalyzed hydrolysis reactions. Therefore, after adding DA, the increase of crystallinity also greatly weakened the degradation ability, so the degradation ability was almost lost when the addition of DA reached 1%.

In order to explore the changes in surface morphology after degradation, AFM scanning was carried out front and back polyester degradation. Before degradation, the sample surface was relatively smooth, as shown in Fig. 10. It can be seen from the image that with the addition of DA, the roughness of the sample surface is significantly reduced. At the same time, the degree of roughness of the water degradation experiment is smaller than that of enzyme degradation. When the addition amount of DA reaches 1%, the roughness of the sample reaches the minimum, and almost no degradation can occur. The AFM results were consistent with the degradation results above and are currently reported in other systems [45–47]. This work has explored a new pathway to tackle the current challenges in composite synthesis reported [48–52] and has the potential to be applied in wide applications not limited to polymer synthesis, nanomaterial synthesis, and material characterization.

4 Conclusions

Using 1,10-decylldiamine as the fourth monomer, PBSeT copolyester reinforced by hydrogen bond was prepared through two-step esterification and one-step polycondensation with excellent mechanical properties and a good vapor barrier. The results of $^1$HNMR and ATR-FTIR
spectra strongly proved the successful introduction of the amide group. The modified PBSeT co-polyethylene had the best comprehensive properties when the content of 1,10-decyldiamine was 0.6% and preserved certain biodegradability. Due to the formation of hydrogen bonds, the intermolecular force is increased and the chain stacking is closer. Thus, the crystallinity is increased by 5.91%. The tear strength, yield strength, and Young’s modulus were increased by 27.3%, 24.1%, and 36.3%, respectively.

Excellent thermal stability at 300 °C ensures the processing performance of copolysters in industrial production. The introduction of the amide group makes copolysters have higher hydrophilicity and also improves the water vapor barrier to a certain extent. It is important to improve the degradability and performance of compound agricultural film and food packaging film. Therefore, aliphatic aromatic copolysters with 1,10-decyldiamine as additive monomer can be a new kind of membrane material. Future research should focus on obtaining higher crystallinity and biodegradability.

Author contribution Y.L. and Z.W. have designed this project and contributed to the main manuscript text. Y.F. conducted experiments and wrote the main manuscript text. Ab.Al., X.M., A. Alh., X.Y., M.I., X.G., H.A., M.H., and W. W. have contributed to conducting the experiments, preparing figures, and writing. All authors reviewed the manuscript.

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Declarations

Competing interests The authors declare no competing interests.

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