

High Damping and Mechanical Properties of Hydrogen-bonded Polyethylene Materials with Variable Contents of Hydroxyls: Effect of Hydrogen Bonding Density*

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Abstract Hydrogen bonding is considered to have significant effect on the interaction between polymeric chains and on the viscoelasticity of the polymeric materials. In this paper, we attempt to discuss the relationship between hydrogen bonding density and damping behavior and mechanical properties of polyethylene-based polymeric materials. For this reason, a series of pendant chain hydrogen bonding polymers (**PCHBP**) with different hydrogen bonding density (HBD) were prepared by quantitatively changing the content of pendent hydroxyl groups on the main chain of polyethylene. It was found that **PCHBP** with low HBD showed similar properties to polyethylene, indicating that the property of the materials was dependent mainly on the structure of the main chain. However, **PCHBP** with high HBD exhibited two $\tan\delta$ peaks and a platform of loss modulus as well as a high storage modulus (about 400 MPa) at the second $\tan\delta$ peak temperature, demonstrating that a polymeric material with high strength and damping properties was obtained. More importantly, the maximum of loss modulus showed a linear increase with the HBD, indicating that a higher HBD greatly improved the damping properties of the polymeric materials.

Keywords Hydroxyls; Polyethylene; Hydrogen bonding; Polymer damping

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INTRODUCTION

Noise and mechanical vibration arising from the operation of machinery, equipment, and vehicles in the environment have been a serious problem since excessive vibration can cause damage either to the surroundings or to the source, and it can also affect peoples' health. Polymeric materials, due to their good viscoelastic behavior and tunable impact properties, have been extensively used as damping materials^[1–5]. Owing to the abundant availability and high mechanical strength, polyethylene has become the most widely used polymer material. Nevertheless, it has also been reported that the damping behavior of polyethylene is not good when used as damping materials^[6–8]. The damping performance of a polymeric material is determined mainly by its internal friction^[9–14], and most of the polymeric materials show a high damping performance around their glass

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transition. However, there is a challenge that the strength of the materials descends so intensively in this region that the material is too flexible to be used for practical applications^[15, 16].

In order to solve the problem, hydrogen bonding interaction is usually introduced into the polymer materials to promote the internal friction and to enhance the polymer strengths^[17–20]. Hydroxyl group, with little steric hindrance and highly directional^[21–24], is the most common side group to provide hydrogen bonding without other uncertainties^[25, 26]. Compared to the long polymer chains, hydroxyl has a small volume that the steric hindrance effect can be neglected^[23, 24, 27]. Polyethylene has the simplest polymeric structure and poor damping behavior so that the interference among the polymeric chains could be ignored^[6–8]. In this work, we choose this simple polyethylene as the main chain, and hydroxyl as the pendant groups to prepare a series of polymers with pendant chain hydrogen bonding (**PCHBP**) to study the influence of hydrogen bonding on the damping properties of polyethylene, aiming to obtain a polyethylene material with high damping behavior and mechanical properties.

The effect of hydrogen bonding to the viscoelastic properties of polymer has been studied extensively. However, quantitative studies on the relationship between hydrogen bonding and viscoelastic properties have been rarely reported and the material with high damping performance and mechanical properties has been hardly obtained^[10, 28–30]. In this paper, we concentrate on a quantitative study to investigate the influence of hydrogen bonding quantity on the viscoelasticity of the **PCHBP** samples. Dynamic mechanical analysis was employed to characterize the dynamic mechanical and thermal properties, including storage modulus (E'), loss modulus (E'') and glass transition temperature (T_g), etc. A combination of different measures was employed to find out the effects of hydrogen bonding on the viscoelastic properties.

EXPERIMENTAL

Materials

Poly(vinyl alcohol-co-ethylene) (**a**, **b**) and poly(ethylene-co-vinyl acetate) (**c**, **d**) were purchased from Sigma-Aldrich. These two polymers were prepared from a similar batch, except the subsequent alcoholysis. Toluene and methanol were obtained from Tianjin Kermel Chemical Reagent Co., Ltd. Sodium hydroxide was purchased from Sinopharm Chemical Reagent Co., Ltd. All these reagents were analytical reagents and used as received. Poly(vinyl alcohol) (PVA, degree of hydrolysis: 99%–100%) was purchased from Sinopharm Chemical Reagent Co., Ltd.

Preparation of Samples with Different Hydroxyl Density

A detailed description of the materials and the synthesis of **PCHBP** are shown in Scheme 1. **PCHBP3** and **PCHBP4** were prepared by melting **a** and **b** and pressed under nitrogen atmosphere to obtain the sample as polymer films. The samples were cooled down and kept at room temperature for 24 h under nitrogen.

PCHBP1 and **PCHBP2** were prepared by alcoholysis of the two samples poly(ethylene-co-vinyl acetate) (**c** and **d**). A toluene solution (10 wt%) was prepared, and a solution of NaOH in methanol (10 wt%) was added dropwise. The products (**e** and **f**) were purified and the sample films were prepared in the same way as for **PCHBP3** and **PCHBP4**.

For comparison, pure PVA sample was also prepared for thermal and mechanical tests. In order to obtain a uniform sample of PVA, an aqueous solution of 10 wt% of PVA was prepared. The solution was cast from a PTFE plate and dried at 60 °C. Then the film was cooled down at room temperature and kept in a moisture-free environment for 24 h.

Instruments

Fourier transform infrared spectroscopy (FTIR)

FTIR of the samples was recorded with a spectrophotometer of Perkin-Elmer (Spectrum 100 FTIR). FTIR spectra at different temperature were acquired on Nicolet 6700 instrument, equipped with Linkam FTIR 600

heating stage. The samples were heated at a heating rate of $10\text{ K}\cdot\text{min}^{-1}$ and kept the temperature constant for 1 min to perform the scanning every $10\text{ }^{\circ}\text{C}$. All the spectra were acquired at a film transmission FTIR method with a resolution of 4 cm^{-1} for 4 scans. No visible signs of degradation were observed during the heating. Duplicate measurements were performed for a number of samples and the reproducibility was good.

Differential scanning calorimetry (DSC)

DSC test was carried out on Q200 Differential Scanning Calorimeter from $-40\text{ }^{\circ}\text{C}$ to the corresponding temperature with a heating rate of $3\text{ K}\cdot\text{min}^{-1}$. All the tests were carried out in aluminum sample pans with vented lids. All the data were recorded from the second scan to erase the previous thermal history.

Dynamic mechanical analysis (DMA)

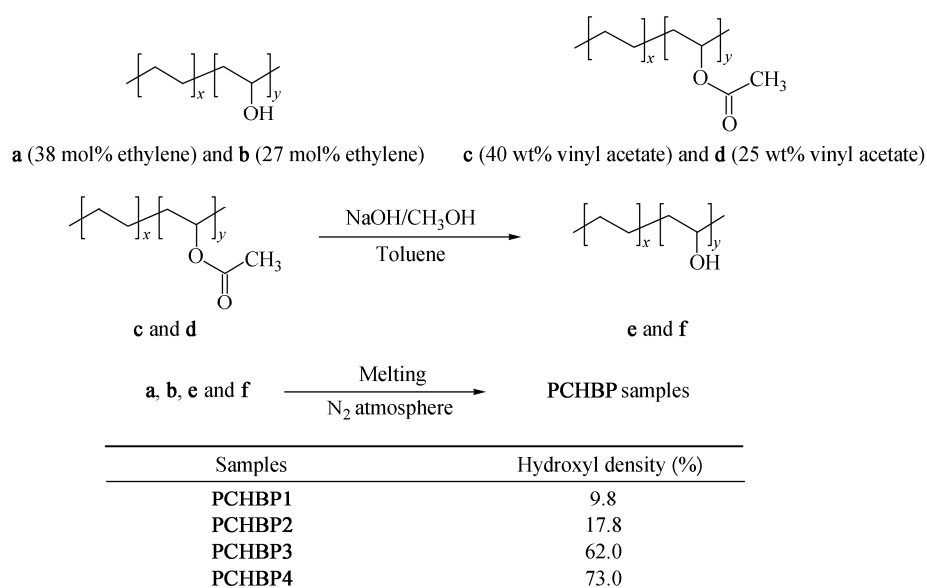
DMA was performed on TA Instruments Q800. The samples were tested in film tensile mode and cooled down to the starting temperature of $-40\text{ }^{\circ}\text{C}$. The DMA test started at $-40\text{ }^{\circ}\text{C}$ and the storage modulus (E'), loss modulus (E'') and the loss factor $\tan\delta$ were measured as functions of temperature. The instrument was operated with controlled sinusoidal force with a frequency and amplitude of 1 Hz and $10\text{ }\mu\text{m}$, respectively, ranging from $-40\text{ }^{\circ}\text{C}$ to the corresponding temperature with a heating rate of $3\text{ K}\cdot\text{min}^{-1}$.

Hot-stage polarized optical microscopy (POM) tests

The transmission and crystalline texture of hydrogen bonding polymer were examined with an Olympus BX51 POM with a Linkam hot stage (LTS350). Samples were heated at a heating rate of $10\text{ K}\cdot\text{min}^{-1}$ and then kept the temperature for 1 min to take the shoot every $10\text{ }^{\circ}\text{C}$. All pictures were taken by Canon EOS 400D digital camera with EOS CAPTURE software.

RESULTS AND DISCUSSION

In this work, four **PCHBP** samples (**PCHBP1–4**) with different contents of hydroxyl groups were chosen to study the influence of the hydrogen bonding on the damping properties (Scheme 1). To demonstrate the results, we define “hydroxyls density” as the molar ratio of the hydroxyls to the repeat units of polyethylene (*i.e.* $-\text{C}_2\text{H}_4-$). We assume that every two hydroxyls form the hydrogen bonding and all hydroxyls contribute to the formation of the hydrogen bonding. Therefore, the “hydrogen bonding density” (HBD) can be defined the same value to “hydroxyls density”. For example, if the “hydroxyls density” is 0.5, the “hydrogen bonding density” is also 0.5.



Scheme 1 Structure and preparation of samples **PCHBP1**, **2**, **3** and **4** with different hydrogen bonding density

Dynamic mechanical analysis was performed to study the relationship between hydrogen bonding and dynamic mechanical properties (including loss factor, modulus and glass transition temperature, *etc.*). According to the theory of polymer viscoelasticity, the dynamic modulus of a polymer in the alternating force field consisted of two fractions, the E' (the storage modulus or the elasticity modulus) and the E'' (the loss modulus or the viscous modulus).

Gel permeation chromatography (GPC) test was performed to study the effect of the molecular weight of the samples. It is very difficult to determine the M_w of **PCHBP3** and **PCHBP4** due to their poor solubility in THF. Considering that the raw materials of poly(vinyl alcohol-*co*-ethylene) (**a** and **b**) were prepared *via* alcoholysis of poly(ethylene-*co*-vinyl acetate), as described by Technical Service of Sigma-Aldrich, it would be reasonable that the M_w and the degree of polymerization would represent the ones of poly(vinyl alcohol-*co*-ethylene).

GPC curves indicated that the raw materials of **c** and **d** showed the similar M_w , **c** about 5.6×10^4 and **d** about 5.4×10^4 (Fig. S1 in supporting information, SI). The number of repeat unit of the samples **c** and **d** was calculated and the M_w values of **PCHBP1** and **PCHBP2** were about 4.5×10^4 and 4.3×10^4 , respectively. It could be also noted from the GPC curves that the molecular weights of raw materials were close to each other and their distributions were narrow, which meant that the M_w and its distribution should have little effect on the mechanical properties of the samples.

The polymer main chains are strongly trammled by the hydrogen bonding formed by —OH groups at low temperature, and the main chain cannot be flexible enough to move freely. It was shown on the curves that samples with high HBD (**PCHBP3** and **PCHBP4**) showed a high storage modulus while the samples with low HBD (**PCHBP1** and **PCHBP2**) showed a relatively lower modulus close to that of the pure polyethylene (Fig. 1a). It is obvious that, although the HBD did not have much differences (from 62% to 73%, 9.8% to 17.8%) the increase in storage modulus from 4611 MPa for **PCHBP3** to 6458 MPa for **PCHBP4** was much greater than that from 1587 MPa for **PCHBP1** to 1766 MPa for **PCHBP2** at about $-40\text{ }^\circ\text{C}$.

When the temperature is below a critical point, there is a strong interaction of hydrogen bonding among polymer chains. Therefore, below the critical temperature, the polymeric materials showed a tough and inflexible behavior, and their properties did not alter intensively with the increasing temperature (Fig. 1a). The storage modulus decreased as the temperature ascended above the critical temperature. Because the intermolecular bonding was gradually destroyed above the critical temperature, the strength of hydrogen bonding was relatively weaker than that at lower temperatures and the movement of the chain segments was much freer. When the temperature was high enough, the strength of hydrogen bonding was destroyed and the material became softened. There was a notable drop in the storage modulus corresponding to the glass transition of the polymer (Fig. 1c, first peak of curves), which was attributed to the movement of chain segments. Moreover, the E' was kept as slight plateau for **PCHBP3** and **PCHBP4**, which indicated that, at higher temperature, the hydrogen bonds were still strong and the polymeric materials were kept tough because of the high hydrogen bonding density. The curves of **PCHBP1** and **PCHBP2** were not recorded above $100\text{ }^\circ\text{C}$ due to the yielding of the samples.

More distinct influence of hydrogen bonding density on the loss modulus and loss factor emerged when the temperature rose up. It is generally accepted that the higher loss modulus is, the better damping properties will be. Loss modulus represents the energy dissipation per unit volume of materials and it is the most intuitive physical parameter to evaluate the damping properties. Figures 1(b) and 1(c) showed the temperature dependence of the loss modulus and the loss factor of the samples, respectively. An interesting observation was that, in the high temperature region (about $120\text{--}150\text{ }^\circ\text{C}$), a loss modulus plateau emerged (**PCHBP3** and **PCHBP4**, Fig. 1b), and at the same time another peak appeared in the curves of loss factor (Fig. 1c). It is well known that two peaks can be observed in DMA measurements in a binary mixture if the components of the mixture are not perfectly miscible. But this result did not fit our work on polyethylene materials with hydrogen bonding. Considering the same main chain structure of the samples, this phenomenon can be related to the change of the amount of hydroxyls and the hydrogen bonding interactions brought by the hydroxyls.

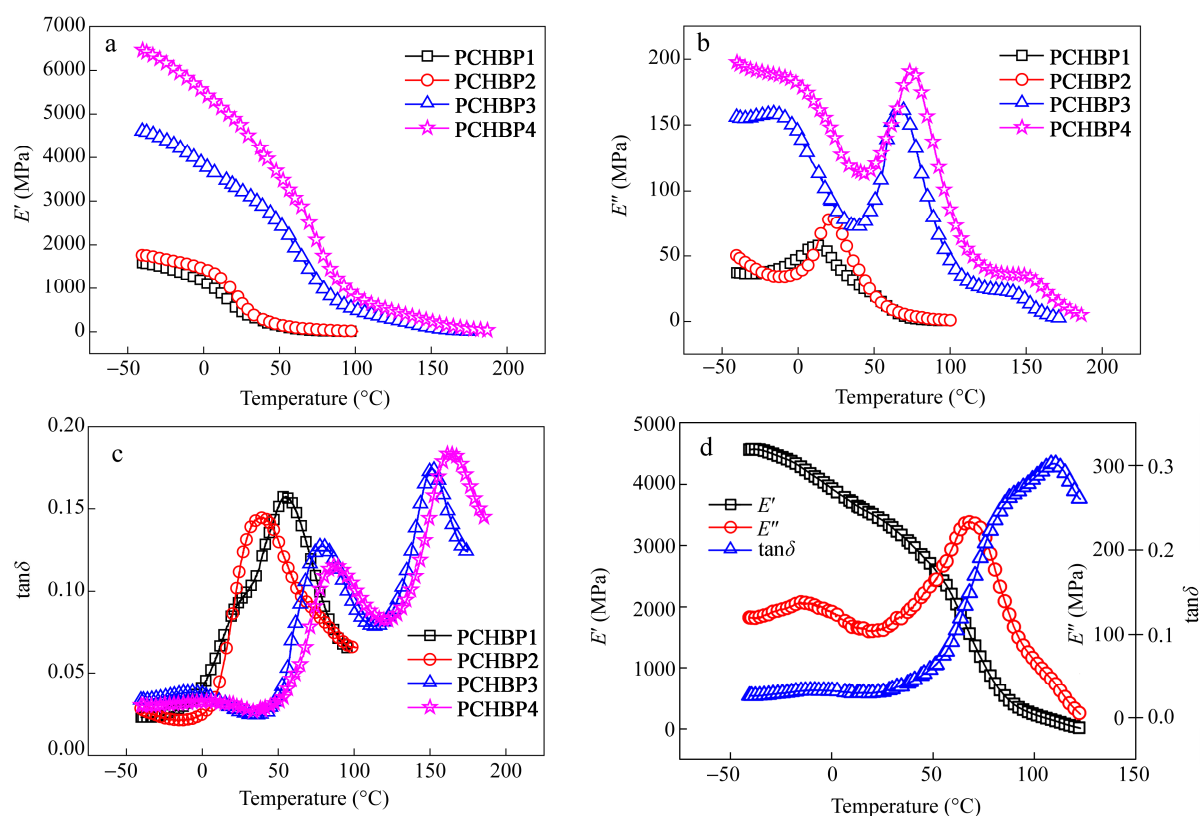


Fig. 1 Temperature dependence of (a) E' , (b) E'' , (c) $\tan\delta$ of PCHBP1 to PCHBP4 with different hydrogen bonding densities and (d) the DMA curves of PVA (99% hydrolyzed)

Figure 2 shows FTIR spectra of different HBD samples. Because of the formation of the hydrogen bonding between H atoms and O atoms of the hydroxyls, the peak position of $-\text{OH}$ with the hydrogen bonding (at about 3340 cm^{-1}) shifted to a lower wavenumber in comparison with that of the free $-\text{OH}$ groups (at about 3650 cm^{-1})^[31]. It was also found that, with the increase in hydroxyl density, the intensity of the $-\text{OH}$ group relative to the methylene peak increased synchronously, demonstrating the relative increase of the hydroxyl amount in another way.

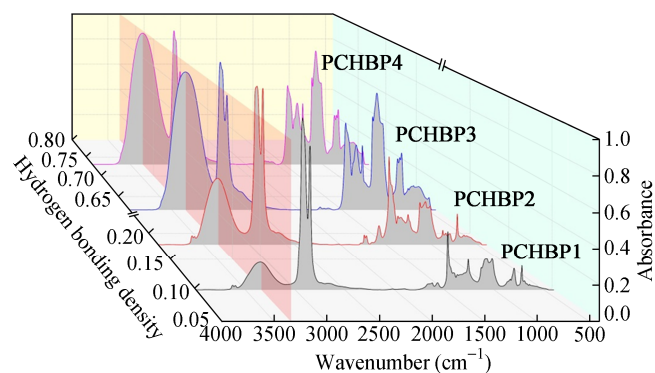


Fig. 2 FTIR studies on the samples PCHBP1, 2, 3 and 4

Plotting the —OH peak position as a function of temperature provides a convenient way to monitor the change in hydrogen bonding with temperature, whereby a shift to higher wavenumber represents a decrease in hydrogen bond strength, and a shift to a lower position means an increase in this strength. The shift of —OH group stretching peak, measured by IR spectroscopy, can indicate the changes of the hydrogen bonding strength as a function of temperature^[32]. Figure 3 shows the variable temperature FTIR spectra of **PCHBP** samples in the region of 3550–3100 cm^{-1} . Because of the low hydrogen bonding density in **PCHBP1** and **PCHBP2**, the temperature of dissociation of the hydrogen bonding was lower than those in **PCHBP3** and **PCHBP4**. As shown in Fig. 3, at glass transition temperature, the bond could vibrate relatively freely in **PCHBP1** and **PCHBP2**, and the bond in **PCHBP3** and **PCHBP4** still kept associated. These relatively free groups can provide additional internal frictions, which is quite important for damping polymeric materials. Otherwise the high hydrogen bonding density of **PCHBP3** and **PCHBP4** made the dissociation difficult to occur at these temperatures, so the main chain of the polyethylene was still bonded distinctly. Although the loss modulus was dependent on the quantity of internal frictions, the value of the storage modulus rose as the hydrogen bonding density increased. Since the decrease of hydrogen bonding strength had more advantages to the decrease of storage modulus, the loss factors of **PCHBP3** and **PCHBP4** were still lower than those in **PCHBP1** and **PCHBP2**.

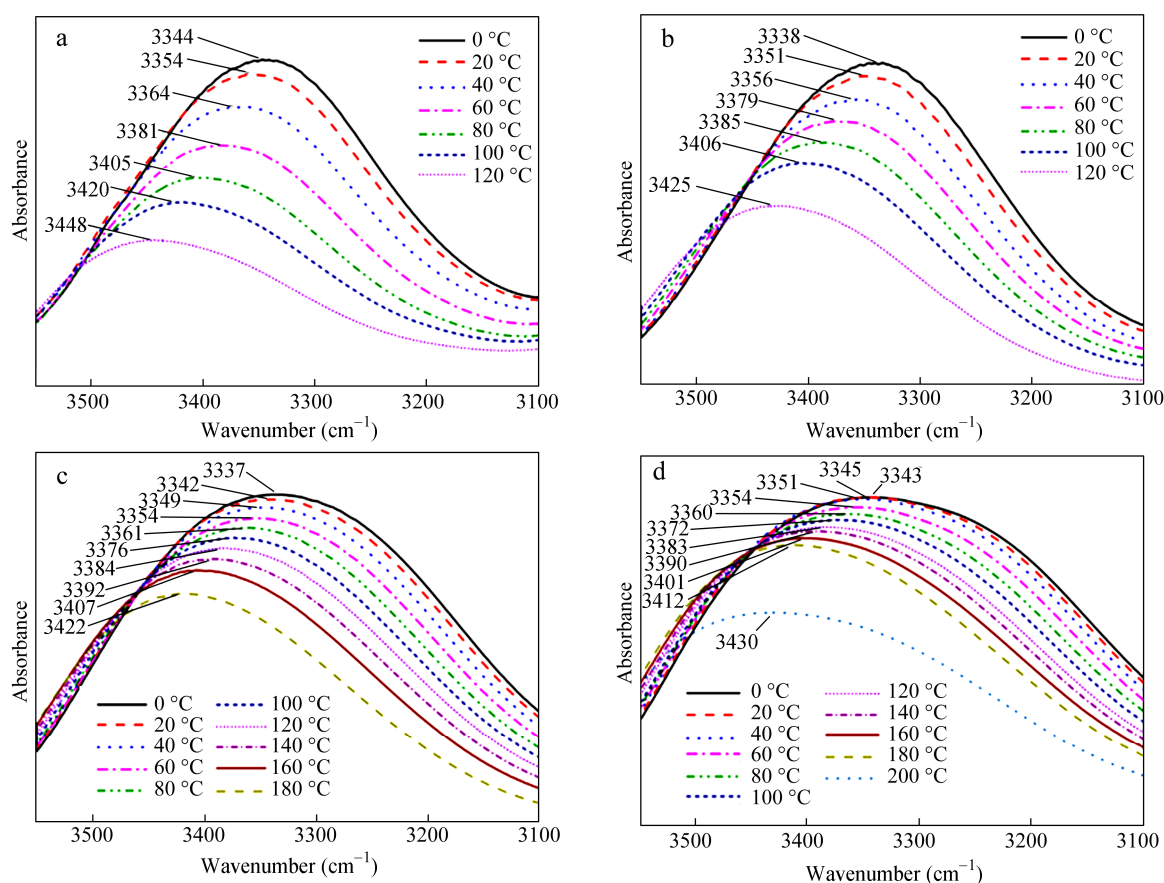


Fig. 3 Variable temperature FTIR spectra of sample **PCHBP1** to **PCHBP4** in 3550 cm^{-1} to 3100 cm^{-1} region (At the glass transition temperature and the second peak range there is a conspicuous change in the frequency of —OH groups: (a) **PCHBP1**, 40–60 $^{\circ}\text{C}$ about 17 cm^{-1} , 60–80 $^{\circ}\text{C}$ about 24 cm^{-1} , (b) **PCHBP2**, 40–60 $^{\circ}\text{C}$ about 23 cm^{-1} , 80–100 $^{\circ}\text{C}$ about 21 cm^{-1} , (c) **PCHBP3**, 80–100 $^{\circ}\text{C}$ about 15 cm^{-1} , 120–160 $^{\circ}\text{C}$ about 23 cm^{-1} and (d) **PCHBP4**, 80–100 $^{\circ}\text{C}$ about 12 cm^{-1} , 140–180 $^{\circ}\text{C}$ about 22 cm^{-1} .)

Polymer with high hydrogen bonding density needs more energy to dissociate the hydrogen bonding, which leads to a higher dissociation temperature observed from variable temperature FTIR spectra (Fig. 3). In Fig. 3, we found that at the glass transition temperatures (**PCHBP1** 40–60 °C, **PCHBP2** 40–60 °C, **PCHBP3** 80–100 °C and **PCHBP4** 80–100 °C) and the second peak range (**PCHBP1** 60–80 °C, **PCHBP2** 80–100 °C, **PCHBP3** 120–160 °C and **PCHBP4** 140–180 °C), there is a conspicuous change in the frequency of —OH groups. It reveals that at these temperatures, the hydrogen bonding between the acceptor and the donor changed distinctly. These temperatures also show the correspondence to the drop of modulus, the plateau modulus and the yielding of the samples. The maximum of the loss factor and the loss modulus at the corresponding dissociation temperature rose as a consequence of more frictions between the polymer chains which were caused by the enhanced mobility of the hydrogen bond donors and the acceptors.

With a combination of dynamic mechanical analysis and variable temperature FTIR spectra, we can assume and it is also reasonable that the hydrogen bonding gradually dissociated with more and more energy input into the system by heating up the samples in this work. The energy distributed to the whole polymeric structures (main chain and side groups), and a sample with higher hydrogen bonding density would show a larger mechanical strength. For a same polymer chain (the same as polyethylene) but different hydrogen bonding density, the glass transition temperature would rise with the hydrogen bonding density.

To further investigate the structural changes upon melting, DSC test and POM observation were carried out for different samples. As shown in Fig. 4, there was a weak step of T_g corresponding to the first peak of loss factor-temperature curves and a sharp peak at higher temperatures (**PCHBP1** 107 °C, **PCHBP2** 107 °C, **PCHBP3** 175 °C and **PCHBP4** 188 °C) corresponding to the second peak (Fig. 1c). The POM images (inserted)

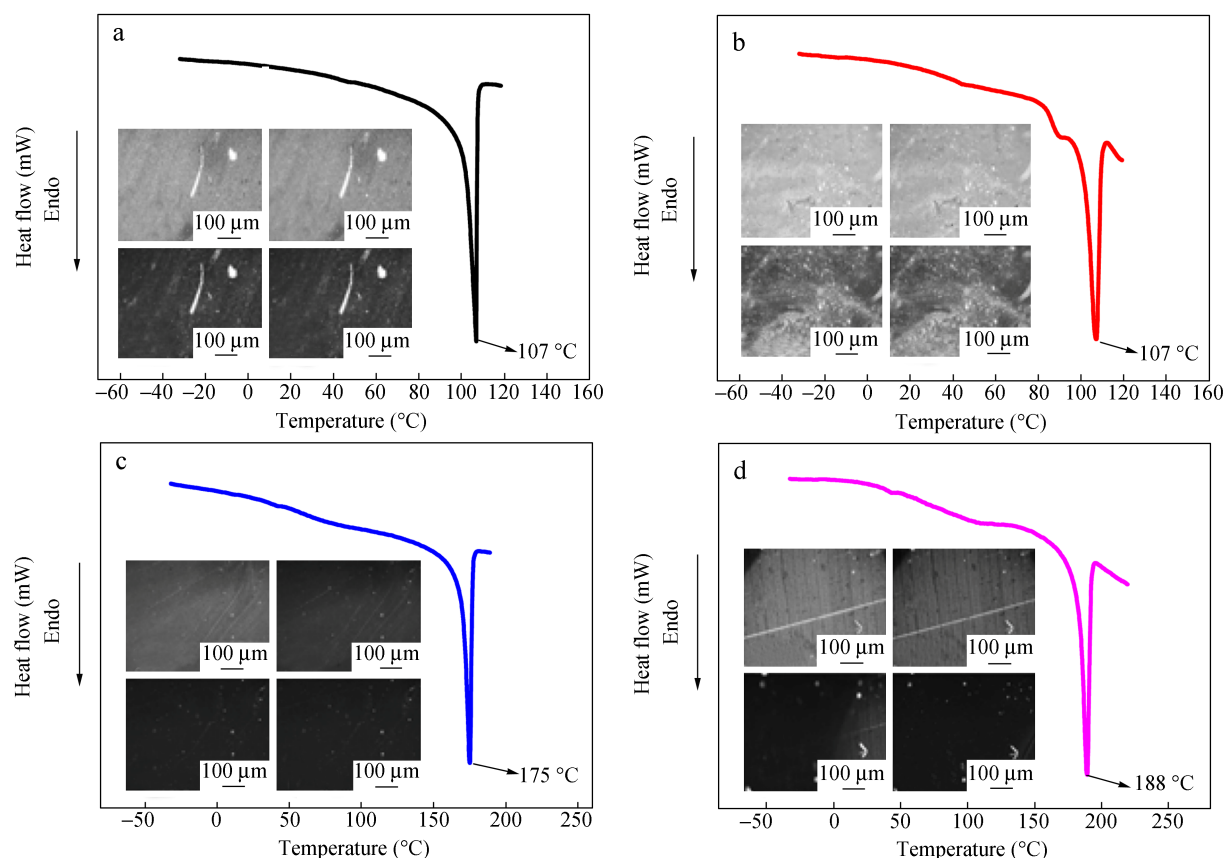


Fig. 4 DSC curves of **PCHBP1**, **PCHBP2**, **PCHBP3** and **PCHBP4** (The inset pictures showed the POM images of the corresponding sample at the melting temperature. The temperature of each POM image (from upper left to lower right, light to dark): (a) **PCHBP1**, 100 °C, 105 °C, 110 °C, 115 °C, (b) **PCHBP2**, 100 °C, 105 °C, 110 °C, 115 °C, (c) **PCHBP3**, 165 °C, 170 °C, 175 °C, 180 °C, (d) **PCHBP4**, 180 °C, 185 °C, 190 °C, 195 °C.)

showed the transition from crystals to amorphous structure and the corresponding transition temperature matched the DSC peak perfectly. The sharp peaks referred to the melting temperature and the change of melting temperature revealed the change of crystal styles. **PCHBP1** and **PCHBP2** showed approximately the same DSC peaks temperature, which were not recorded on Fig. 1 (above 100 °C) due to yielding of the samples at the temperature of about 110 °C^[33]. These temperatures are much lower than the melting temperature of **PCHBP3** and **PCHBP4**. It should be noted additionally that **PCHBP3** and **PCHBP4** showed distinct changes in peak temperature relative to **PCHBP1** and **PCHBP2**, and the peak temperature changed more (from 175 °C to 189 °C) while the hydrogen bonding density changed little (from 62% to 73%) than **PCHBP2** and **PCHBP1** (both are 107 °C).

From the above studies, it can be concluded that the thermal properties of the polymer with a low HBD (such as 9.8%) mainly depended on the main chains and the content of hydrogen bonding was too small to provide great contribution. However, at a high HBD, the hydrogen bonding showed a significant effect on the properties of the hydrogen bonding interaction. It also demonstrated that the high HBD had much more influence on the thermal properties of polymer materials compared to the one with low HBD.

Table 1 shows the FTIR wavenumber of hydroxyl of different samples at T_g and T_m . Surprisingly, it was found that, although the HBD was quite different in the four samples, their T_g , T_m and the resonance peak of hydroxyl showed great similarity at T_g (3364 cm^{-1}) and T_m (3416 cm^{-1}), which indicated that the hydrogen bonding had the same strength under these conditions (Fig. S2 in SI). According to the DSC and POM experiments, the increase of HBD changed the crystallization from the polyethylene with weak van der Waals force between polyethylene chains to the hydrogen bonding induced crystallization with the strong hydrogen bonding interactions.

Table 1 FTIR wavenumber of —OH of **PCHBP1–PCHBP4** at T_g and T_m

Samples	T_g^a (°C)	T_m^b (°C)	Wavenumbers of —OH at T_g (cm^{-1})	Wavenumbers of —OH at T_m (cm^{-1})
PCHBP1	40.3	107	3364	3416
PCHBP2	55.0	107	3364	3416
PCHBP3	79.4	175	3361	3414
PCHBP4	89.0	188	3364	3416

^a Measured by DMA; ^b Measured by DSC

It is known that the glass transition and melting are different motion states of the polymer chains. With the same main chain in the four samples, the motional behavior of polymer chains was regulated and altered by the hydrogen bonding interactions. Because of the same strength of intermolecular force at T_g and T_m , it could also be concluded that the main chains of the polymer had the similar stereoregularity and copolymerizing randomness.

At a low temperature, hydroxyls wrapped in the small crystal grains were tightly trammled and the high loss modulus for **PCHBP3** and **PCHBP4** might originate from the internal frictions provided by the crystal grains. The increase of temperature led to the melting of the grains surface and the weakening of the interaction between the grains, while the hydrogen bonding inside the crystal grains still remained bonded. When the crystal grains began melting, the loss modulus ascended until the peak emerged (Fig. 1b). The plateau modulus of the E'' was observed owing to the crystal grains melting at the temperature about 180–190 °C. The hydrogen bonding interactions hold the polymer chain together (act as “glue”) to keep **PCHBP3** and **PCHBP4** from viscous flow state and to provide relatively high storage modulus for the materials. The hydrogen bonding interactions act as not only the “glue” but also the damping layer between the main chains. At the melting temperature of the polymer, the crystal grains completely melted, and the amount of freely mobile hydroxyls increased to provide more internal frictions. Figure 1(b) shows a plateau loss modulus at the melting point because of the increase in internal frictions with relative sliding of the donor and the acceptor, hence the second loss factor peak is observed in Fig. 1(c). So at this temperature, **PCHBP3** and **PCHBP4** have a high mechanical

strength and also show good damping properties.

In this work, we also tested the dynamic mechanical properties of PVA (99% hydrolyzed) with a hydrogen bonding density of 99%. Because of the high HBD and the excessive high interactions between the polymer chains, the PVA samples showed different properties (Fig. 1d). Considering that there would be moisture remaining in the polymer and the high hydrogen bonding density of the PVA, the polymer showed a relatively low temperature of $\tan\delta$ peak and the sample showed no melting point till its pyrolysis. But the loss modulus of PVA sample still fitted the line relationship because of the high temperature disassociated the hydrogen bonding and promoted the internal frictions.

The dependence of the maximum loss modulus on hydrogen bonding density in different samples is shown in Fig. 5. It was shown that the values of the maximum modulus and the points fit a good linear fashion. It was observed from the line that the maximum of the loss modulus provided by the interactions of hydrogen bonding increased with HBD in a linear manner and we could also assume that the limit value of loss modulus was provided by the interactions of the hydrogen bonding (about 240 MPa).

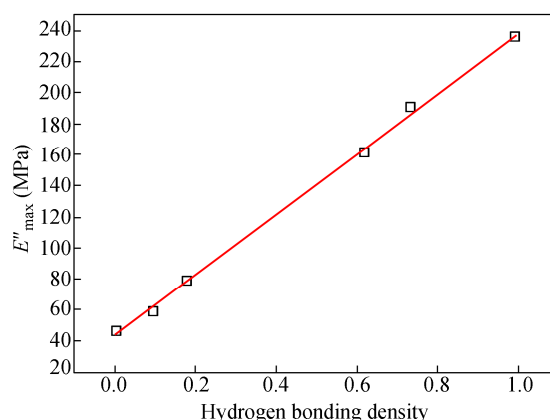


Fig. 5 Maximum loss modulus of samples with different HBD

In order to validate the correctness of the $E''_{\max}$ -HBD line, DMA curves of the pure polyethylene were recorded (Fig. S3 in SI). The curves showed the maximum of loss modulus was about 47 MPa. Considering the inevitable side chains of polyethylene, it fitted the $E''_{\max}$ -HBD line well.

CONCLUSIONS

In summary, damping property was studied by introducing hydrogen bonding into the polyethylene system. Hydrogen bonded polyethylene with low HBD maintained the properties of the polyethylene chains, while the damping properties with high HBD were strongly affected by the interaction of hydrogen bonding. Dramatic promotion of loss factor ($\tan\delta$) was observed with HBD increase, and the modulus also changed distinctly because of the interaction of the hydrogen bonding. The $\tan\delta$ curves of high HBD polymer showed two peaks; one was attributed to the glass transition temperature and the other to the melting point of the polymers. FTIR peak of the hydroxyls group shifted to higher wavenumbers conspicuously at both $\tan\delta$ peaks, and it demonstrated that the hydrogen bonding strength decreased remarkably around this temperature.

The hydrogen bonding in the polyethylene materials acts not only as a “glue” but also as a damping layer between polymer chains. It can provide higher storage modulus and more internal frictions, so the samples with high hydrogen bonding density show both “tough” and “viscous” (damping) performance at a high temperature. These changes can broadly promote the availability of materials on wide temperature ranges and high damping properties. The maximal values of the loss modulus increased with HBD, which provided a new avenue to study and to develop novel damping polymeric materials.

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