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Plasticizing and Strengthening Poly(vinyl chloride) with Structurally Different Ionic Liquids

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Electronic Supplementary Information

Abstract This study simultaneously plasticizes and strengthens poly(vinyl chloride) (PVC) with structurally different ionic liquids (ILs), namely monomeric [Bmim]NTf_2, linear polymerized $PImC_6NTf_2$, and branched polymerized P(VIm-4)NTf_2. Notably, $PImC_6NTf_2$ exhibits the best performance. Dynamic mechanical analysis reveals that the glass transition temperature of $PImC_6NTf_2$/PVC composite film is lower than that of dioctyl phthalate (DOP)/PVC, indicating enhanced processability. Compared with neat PVC film, the 4% $PImC_6NTf_2$/PVC film exhibits 747.2%, 165.4%, and 49.1% increases in elongation at break, tensile strength, and Young's modulus, respectively, along with a 27.4-fold rise in fracture energy. Additionally, the thermal stability, hydrophobicity, and wear resistance of PVC are also improved. These findings demonstrate the great potential of linear polymerized ILs as innovative PVC plasticizers.

Keywords Poly(vinyl chloride); Ionic liquid; Plasticizer; High toughness; High strength

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INTRODUCTION

Since its commercial production commenced in 1930, poly(vinyl chloride) (PVC) has emerged as a fundamental material across a wide range of industries including construction, packaging, automotive, medical, toy manufacturing, and coatings. Its widespread adoption is attributed to its outstanding properties such as high mechanical strength, excellent corrosion resistance, inherent flame retardancy, and cost-effectiveness.[1–6] It holds the second position in global plastic usage.[7] However, the inherent rigidity of PVC necessitates the addition of plasticizers to improve its flexibility and processability.[8,9] Phthalate esters, which account for 87% of the market, remain the dominant choice.[10] Despite their widespread application, these plasticizers exhibit several drawbacks, including limited compatibility, poor stability, reduced lubrication at low temperatures, and a tendency to migrate and precipitate.[11–13] These limitations significantly hinder their use in outdoor engineering plastics.

Researchers are actively exploring alternative plasticizers with enhanced performance and environmental compatibility. Fu *et al*.[14] synthesized a range of castor-oil-based plasticizers and found that epoxy-acetylated castor oil outperformed dioctyl terephthalate (DOTP) and epoxidized soybean oil (ESO) in terms of tensile strength, migration resistance, solvent resistance, and thermal stability. Liu *et al*.[15] synthesized an eco-friendly plasticizer derived from lactic acid, acetylated 1,4-cyclohexane dimethyl lactate (ALCH), and evaluated its impact on the properties of PVC. The research demonstrated that ALCH increases both the elongation at break and the tensile strength of PVC. These new plasticizers enhance the processing performance of PVC, mitigate the issues associated with phthalic acid esters, and, to a certain extent, bolster the mechanical strength.

Ionic liquids (ILs), composed of organic cations and organic/inorganic anions,[16,17] are well known for their low flammability, low volatility, high thermal stability, and good chemical stability.[18–21] The physicochemical and biological properties of ILs, such as polarity, hydrophilicity, viscosity, and conductivity, can be precisely tailored by modifying their anionic and cationic components.[22,23] ILs with different structures may have different effects on the properties of polymers. Mustafizur *et al*.[24] found that imidazolyl and phosphoryl ILs can be used as effective plasticizers for PVC. These ILs not only provided good plasticizing effects but also exhibited excellent thermodynamic compatibility. Ahmed *et al*.[25] added a specific cyanoimidazolium bromide IL to nylon 6 (PA6) fibers, forming hydrogen bonds between the IL and PA6 molecules, to obtain high-strength PA6 fibers. Plamus *et al*.[26] examined the impact of ILs on the mechanical properties of electrospun polyacrylonitrile membranes, revealing that the specific

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stress was markedly increased with the addition of [Emim]Br, while the use of [Bmim]Cl in DMF resulted in the specific stress of the membrane reaching the most stable state.

Liu *et al*. successfully prepared PVC/dibutyl adipate (DBA) /IL gels using DBA as a plasticizer and incorporated small amounts of imidazolium-type ILs. The PVC/DBA/IL gels exhibited significant improvements in bending displacement, output stress, and pressure sensitivity.[27] Li *et al*. studied the effects of imidazolium ILs on the structure and electromechanical properties of PVC/DBA gels. The results showed that longer cation alkyl chains significantly enhance the dielectric constant, bending performance, and pressure sensitivity.[28]

Furthermore, poly(ionic liquids) (PILs) represent a distinctive category of ILs. Their molecular architecture incorporates both anionic and cationic groups derived from IL monomers,[29–31] endowing them with the advantageous characteristics of ILs while retaining the beneficial properties of conventional polymers. This unique combination renders PILs highly versatile and functionally superior.[32] PILs exhibit unique temperature-responsive behaviors: they are liquid at elevated temperatures, where they function as effective plasticizers, and vitrify into a rigid glassy state at room temperature, thereby significantly enhancing the mechanical strength of the polymers. This dual-phase characteristic makes PILs highly versatile for applications that require both flexibility and structural integrity. Guo *et al*.[33] synthesized a polyether imidazolium IL (PIIL) to modify polypropylene, which led to a notable enhancement in its crystalline structure and mechanical properties, including its tensile strength and toughness. This advancement suggests that by strategically tailoring the structure of ILs, it is possible to develop IL-based plasticizers that not only provide effective plasticization, but also improve the mechanical performance of PVC.

In this study, three structurally different ILs were employed as plasticizers for PVC: monomeric IL [Bmim]NTf_2, linear PIL $PImC_6NTf_2$, and branched PIL P(VIm-4)NTf_2. PVC composite films incorporating these ILs were prepared using the solution casting method, and their properties were systematically compared with those of films plasticized with the traditional plasticizer di(2-ethylhexyl) phthalate (DOP).

EXPERIMENTAL

Materials

DOP (99%) was purchased from Sinopharm Chemical Reagent Co., Ltd. (Fig. 1a). 1-Butyl-3-methylimidazolium bis(trifluoromethanesulfonyl)imide ([Bmim]NTf_2, 98%, Fig. 1b), lithium hydride (LiH, 97%), 1-bromo-6-chlorohexane (98%), imidazole (99%), and 2,2′-azobis(2-methylpropionitrile) (AIBN, CP) were purchased from Meryer (Shanghai) Chemical Technology Co., Ltd. Lithium bis(trifluoromethanesulphonyl)imide ($LiNTf_2$, 99%), 1-vinylimidazole (VIm, 99%), ethyl acetate (99%) and 1,2,4,5-tetrakis(bromomethyl)benzene (TBMB, 98%) were purchased from Shanghai Macklin Biochemical Technology Co., Ltd. Tetrahydrofuran (THF, AR), *N*,*N*-dimethylformamide (DMF, AR), dimethyl sulfoxide (DMSO, AR), dichloromethane (DCM, AR), ethylene glycol (EG, AR), and methanol (AR) were purchased from Sinopharm Chemical Reagent Co., Ltd. PVC (SG-5) with a K value of 66–67, molecular weight of 62500–68100 g/mol, particle size of 63–250 μm, original whiteness of 93%, and apparent density of 0.525–0.555 g/cm^3. No chemical reagents were used for purification.

Synthesis of PILs

The $PImC_6NTf_2$ and P(VIm-4)NTf_2 PILs were synthesized by free radical polymerization, as shown in Figs. 1(c)[34] and 1(d)[35]. To synthesize $PImC_6NTf_2$, 3.17 g of imidazole was dissolved in THF (10 mL) and LiH, while 0.45 g of LiH was dispersed in another 10 mL of THF. Both the solutions were cooled to 0 °C under continuous stirring. The imidazole solution was then slowly added to the LiH mixture under nitrogen atmosphere, maintaining the temperature at 0 °C. Subsequently, 5.39 g of 1-bromo-6-chlorohexane was gradually introduced and the reaction was stirred for 24 h under nitrogen protection. After completion of the reaction, 10 mL of water was added to quench the reaction. The upper organic layer was collected using a pipette, and THF was removed using a rotary evaporator. The residue was dissolved in deionized water and the product was extracted with DCM. Following phase separation, the DCM layer was collected and concentrated *via* rotary evaporation. This extraction process was repeated thrice to obtain ImC_6Cl. For polymerization, 3.73 g of ImC_6Cl was dissolved in 2 mL of ethylene glycol and reacted at 90 °C for 12 h. After cooling, 10 mL of methanol was added to dissolve the product, and the solution was added dropwise to 200 mL of acetone to precipitate a white solid. The precipitate was sequentially washed with acetone and ethanol for purification and then dried to obtain $PImC_6Cl$. Finally, 1 g of $PImC_6Cl$ was dissolved in 50 mL of water, followed by the addition of 100 mL of 0.35 mol/L $LiNTf_2$ aqueous solution for 12 h ion exchange. The resulting product was separated by centrifugation, washed thrice with water, and vacuum-dried at 80 °C for 12 h to obtain $PImC_6NTf_2$.

The synthesis of P(VIm-4)NTf_2 was carried out as follows: 1.8 g of TBMB was dissolved in 20 mL of THF under an ice-water bath and nitrogen protection, with stirring until fully dissolved. Then, 4.42 g of VIm was then added to the solution, and stirring was continued for 30 min at room temperature. The ice bath was removed and the mixture was stirred at room temperature for an additional 30 min. The mixture was heated to 60 °C and the reaction was allowed to proceed for 12 h under nitrogen protection. After the reaction, the precipitated product was washed five times with ethyl acetate. Each wash was followed by centrifugation to separate the precipitate from the supernatant, which was then discarded. The product was then vacuum-dried at 70 °C for 6 h. Next, the dried product was dissolved in 40 g of water and 9 g of $LiNTf_2$ was dissolved in another 40 g of water. The $LiNTf_2$ solution was slowly added to the product solution and the mixture was stirred at room temperature for 6 h. After stirring, the precipitate was collected and washed four times with deionized water. The resulting intermediate monomer VIm-4NTf_2 was obtained after drying. To proceed with polymerization, 1.5 g of VIm-4NTf_2 was dissolved in 7 mL of DMSO and 1 mL of water, and 0.2 g of AIBN was added. The mixture was stirred for 2 h before being transferred to a 50 mL hydrothermal reactor, where it was reacted at 100 °C for 24 h. After the reaction, the solvent was removed *via* rotary evaporation. The crude product was sequentially washed with deionized water and ethanol, followed by vacuum drying at 70 °C for 12 h to yield P(VIm-4)NTf_2.

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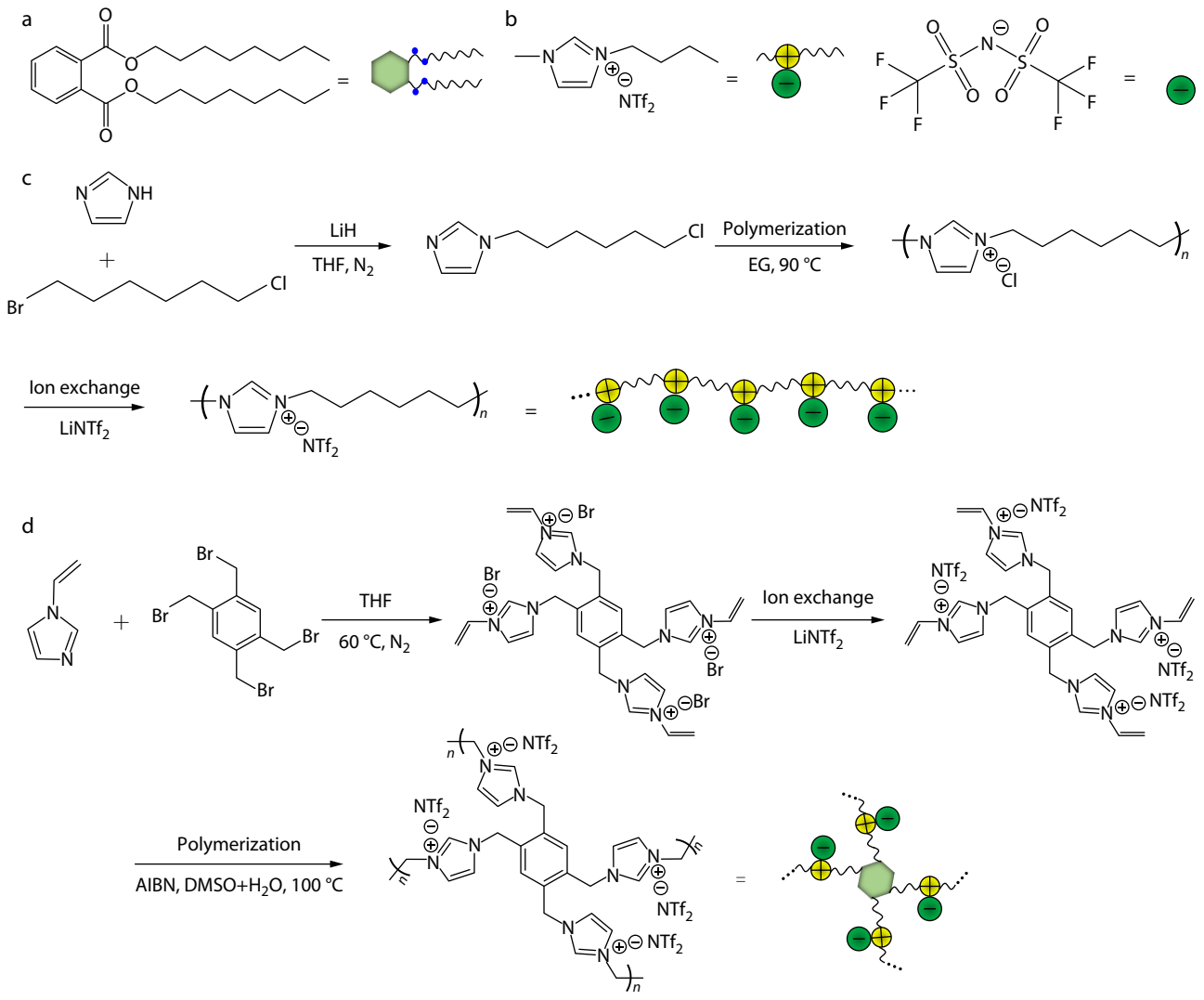

Fig. 1 Chemical structures and simplified schematics of (a) DOP and (b) [Bmim]NTf_2, and synthesis schemes of (c) $PImC_6NTf_2$ and (d) P(VIm-4)NTf_2.

Preparation of PVC Composite Films

The PVC composite films were prepared using the solution-casting method, as illustrated in Fig. 2. First, 60 g of PVC powder was dissolved in 540 g of DMF and heated in a water bath at 60 °C for 1 h to obtain a 10 wt% PVC/DMF solution. Next, 99 g of the solution was aliquoted, (P)ILs (0.1 g) or DOP were added to the solution, and the mixture was stirred for 1 h to ensure uniform dispersion of the additives, resulting in an IL (or DOP)/PVC/DMF mixed solution. Finally, the mixed solution was slowly poured into a 9 cm × 9 cm glass mold and vacuum-dried at 55 °C for 24 h. After drying, the PVC composite films were carefully removed from the mold to obtain 1 wt% IL (or DOP)/PVC composite films. The same procedure was followed to prepare films with different additive concentrations (2%, 3%, 4%, and 5%).

Characterizations

The ^{1}H-NMR spectra of two PILs' intermediates, ImC_6Cl and VIm-4NTf_2, were recorded on an NMR spectrometer (Bruker Avance III 400 MHz, Bruker Co., Ltd., Germany). Fourier transform infrared spectroscopy (FTIR, Bruker RQUINOX55, Bruker Co., Ltd., Germany) was used to identify the functional groups and chemical bonds in the PILs. Attenuated total reflectance FTIR (ATR-FTIR) was used to determine the enhancing mechanism by incorporating (P)ILs from the perspective of inter-molecular interactions, for example, hydrogen bonding and electrostatic interactions. Both FTIR and ATR-FTIR were performed with a resolution of 4 cm^{-1} in the wavenumber range of 500–4000 cm^{-1}. To verify the dispersion of (P)ILs in the PVC composite films, energy-dispersive X-ray spectroscopy (EDS) mapping was performed using a scanning electron microscope (SEM, S-4800, Hitachi, Japan) to check the distribution of characteristic elements in (P)ILs, that is, S, F, and N. The thermal stability of the films was analyzed using a simultaneous thermal analyzer (TGA, NETZSCH 449F5 STA, NETZSCH Group, Germany) under nitrogen flow, with a temperature range of 30–800 °C and a heating rate of 10 °C/min. The contact angles of the films were measured using an SDC-350 optical contact angle measuring instrument (Dongguan Shengding Precision Instrument Co., Ltd., China). Dynamic mechanical analysis (DMA) was conducted using a DMA Q800 instrument (TA Instruments Inc., USA) with a fixed frequency of 1 Hz, temperature range of 30–160 °C, and linear

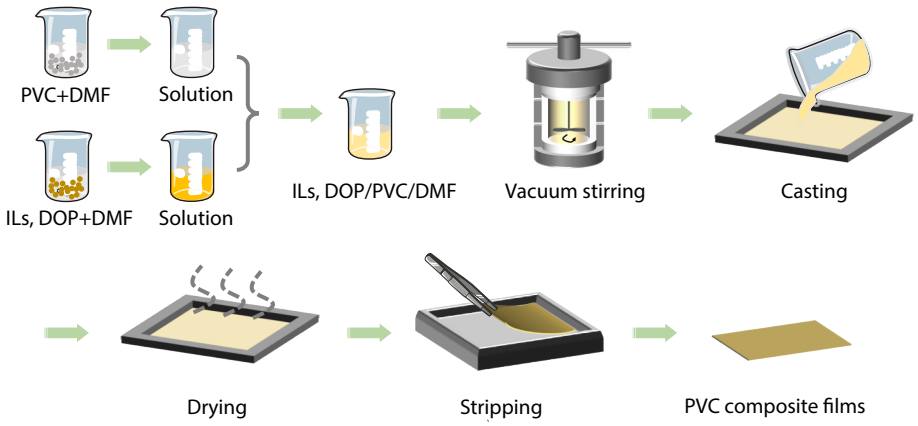

Fig. 2 Preparation process of PVC composite films.

heating rate of 10 °C/min. The surface conductivities of the films were evaluated using a four-point probe detector (RTS-9, Guangzhou 4Probes Technology Development Co., Ltd., China).

The mechanical properties of the PVC composite films were tested following the GB/T 9341-2008/ISO 178:2001 standard, using a 'dumbbell' spline specimen (width: 4 mm, gauge length: 25 mm) at a tensile rate of 10 mm/min on a Shanghai New Sansi C42.104 electronic universal testing machine. At least five tests were performed to obtain the average values of the tensile strength, elongation at break, and Young's modulus. The fracture surface morphology of the films was observed using SEM at an accelerating voltage of 5 kV. Migration tests were conducted according to ASTM D1239-98, using ethanol and n-hexane. PVC films measuring 20 mm × 10 mm × 0.05 mm were immersed at 50 °C for 24 h. Before immersion, the samples were vacuum-dried at 50 °C for 12 h and weighed to obtain the initial weight W_0. After soaking, the films were rinsed with ethanol, vacuum-dried at 50 °C to a constant weight, and re-weighed to obtain the final weight W_1. The percentage weight loss was calculated using the following formula, where W_0 is the initial weight, and W_1 is the final weight of the PVC specimen.

$$\text{Weight Loss } (\%) = \left(\frac{W_0 - W_1}{W_0}\right) \times 100\% \quad (1)$$

Because (P)ILs are novel and effective lubricants, they would probably improve the tribological properties of PVC, which are important for the service time of PVC materials. Therefore, the tribological properties of the composites were evaluated using a multifunctional material surface performance tester (CFT-I, Lanzhou Zhongke Kaihua Technology Development Co., Ltd., China) in ball-on-disk mode. A 100Cr6 steel ball (diameter 4.9 mm, surface roughness (R_a) <30 nm) was used as the counterpart under reciprocating motion, and the ball was cleaned using ultrasonic alcohol treatment before use. The tests were conducted under a normal applied load of 5 N with an average reciprocating distance of 3 mm for 30 min at room temperature. To minimize experimental errors, three repeated tribological tests were performed.

RESULTS AND DISCUSSION

Characterization of PILs

The ^{1}H-NMR spectra of ImC_6Cl and VIm-4NTf_2 are shown in Figs. S1 and S2 (in the electronic supplementary information, ESI), where the characteristic peaks agreed well with the references.[34,35] For ImC_6Cl: ^{1}H-NMR (400 MHz, $CDCl_3$, δ, ppm): 7.45 (s, 1H), 7.03 (s, 1H), 6.91 (s, 1H), 3.94 (t, J=7.1 Hz, 2H), 3.51 (t, J=6.5 Hz, 2H), 1.85–1.69 (m, 4H), 1.52–1.40 (m, 2H), 1.37–1.24 (m, 2H).[34] For VIm-4NTf_2: ^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): 9.39 (s, 4H), 8.27 (t, J=1.9 Hz, 4H), 7.73 (t, J=1.9 Hz, 4H), 7.41 (s, 2H), 7.29 (dd, J=15.6, 8.7 Hz, 6H), 5.98 (dd, J=15.6, 2.5 Hz, 4H), 5.59 (s, 8H), 5.50 (dd, J=8.7, 2.5 Hz, 4H).[35] Fig. 3(a) shows the FTIR spectra of PImC_6NTf_2 and P(VIm-4)NTf_2. Through a detailed analysis of the characteristic absorption peaks, the presence of the anion NTf_2^- was confirmed as follows: the bands at 571 and 738 cm^{-1} were assigned to the shear vibration of the —CF_3 group, the band at 790 cm^{-1} corresponds to the stretching vibration of the —CS group, the bands at 1131 and 1350 cm^{-1} are associated with the stretching vibration of the O=S=O group, and the bands at 654 and 1060 cm^{-1} correspond to the shear and symmetric stretching vibrations of the —SNS group, respectively.[36,37] Furthermore, the —CN group in the imidazole ring exhibited characteristic stretching vibration bands at 1384 and 1566 cm^{-1}, while the bands at 2848 and 2920 cm^{-1} were indicative of the stretching vibration of the —CH_2 group, and the band at 1469 cm^{-1} was attributed to the shear vibration of the —CH, —CH_2, and —CH_3 groups.[37]

Fig. 3(b) shows the TG curves of PImC_6NTf_2 and P(VIm-4)NTf_2. The decomposition onset temperature, a key indicator of thermal stability, is primarily determined by the chemical structure of the PILs' main chains.[32] The initial decomposition temperature of PImC_6NTf_2 was as high as 375 °C, while P(VIm-4)NTf_2 decomposed continuously from approximately 80 °C, which indicates that PImC_6NTf_2 has better thermal stability than P(VIm-4)NTf_2. This difference is primarily attributed to the branched structure of P(VIm-4)NTf_2, which increases the length of the alkyl chain between the polymer main chain and ionic substituent, leading to reduced thermal stability.[38]

Characterization of PVC Composite Films

EDS mapping images of the (P)IL/PVC films are shown in Fig. S3 (in ESI). The characteristic elements of (P)ILs (S, F, and N) are uniformly distributed across the films, confirming good dispersion of (P)ILs. The TG curves of the PVC composite films are shown in Fig. 4(a). PVC has two decomposition stages; the initial stage is important for its thermal stability. The initial stage is primarily

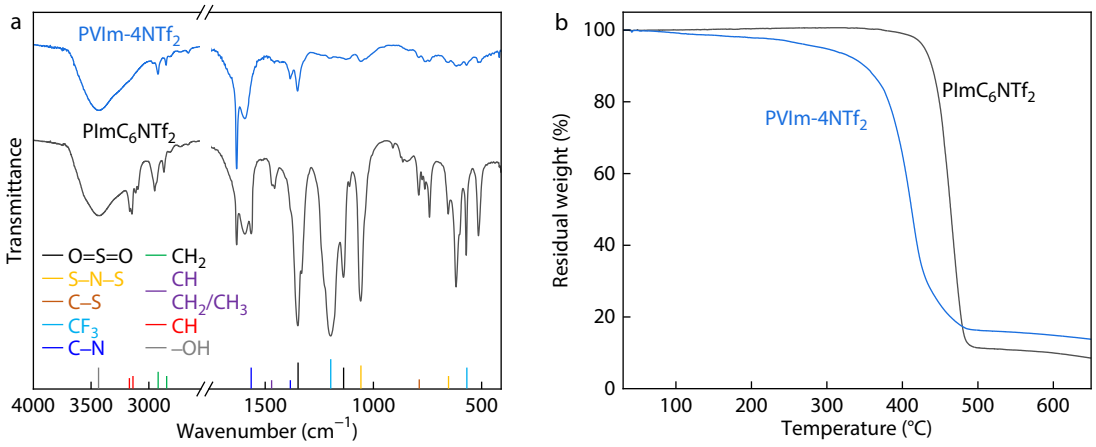

Fig. 3 (a) FTIR spectra and (b) TG thermograms of $PImC_6NTf_2$ and $P(VIm\text{-}4)NTf_2$.

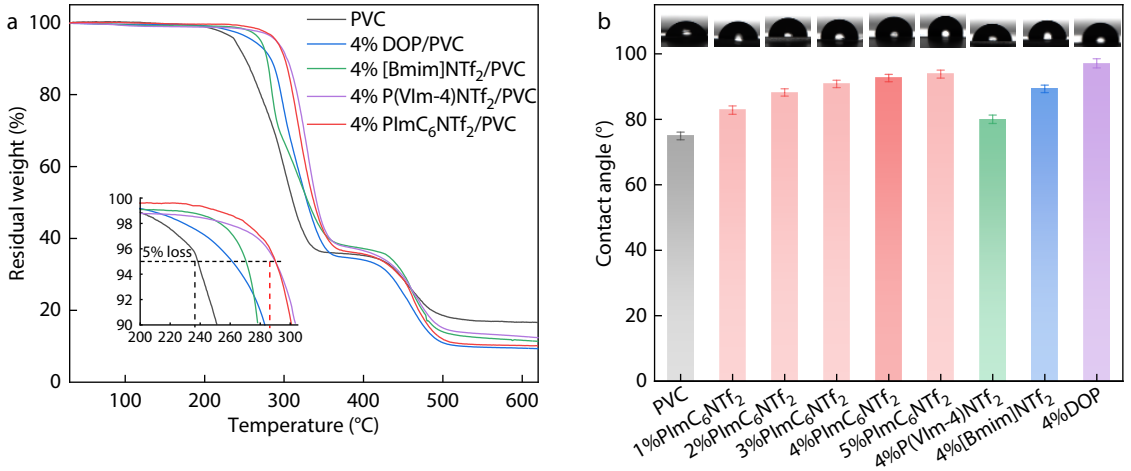

Fig. 4 (a) TG thermograms and (b) contact angles of different PVC composite films.

attributed to the dehydrochlorination reaction, and the second stage involves the thermal breakdown of the PVC main chain.[39,40] PVC composite films containing (P)ILs started to decompose at higher temperatures than neat PVC films, especially for the two PILs. From the enlarged TG curves, the temperatures corresponding to 5% weight loss for the films are as follows: PVC at 238 °C, 4%DOP/PVC at 261 °C, 4%[Bmim]NTf_2/PVC at 270 °C, and 4%P(VIm-4)NTf_2/PVC and 4%$PImC_6NTf_2$/PVC at 290 °C. The improved thermal stability by (P)ILs incorporation was primarily due to the formation of hydrogen bonds between the (P)ILs and PVC chains, which hindered the breakage of the PVC chains.[41] The pronounced improvement by PILs is primarily attributed to the conjugated structure and steric hindrance effects[38] as well as the inherent high thermal stability of the PILs themselves.[42]

Fig. 4(b) illustrates the variation in the contact angle of the PVC composite films. As the $PImC_6NTf_2$ content increased, the contact angle of the composite film also increased, indicating an enhancement in the hydrophobicity of the film. Furthermore, the contact angles of the PVC composite films containing 4%(P)ILs and DOP were compared. The contact angle of the $PImC_6NTf_2$/PVC film was higher than that of the other two (P) IL-incorporated PVC films and only slightly lower than that of the DOP/PVC film. This is mainly due to the higher hydrophobicity of $PImC_6NTf_2$. The hydrophobicity of (P)ILs increases with the length of the alkyl chains on their cations, whereas the hydrophilicity or hydrophobicity of the anions is determined by their coordination abilities.[43] The higher hydrophobicity of $PImC_6NTf_2$ is primarily attributed to the presence of longer alkyl chains.[44,45]

Additionally, it is noteworthy that all of the (P) IL/PVC composite films maintained excellent insulating properties, with resistivity values exceeding the upper limit of the RTS-9 double electric measurement four-point probe tester (Guangzhou 4Probes Technology Development Co., Ltd., China, measurement range: 10^{-5}–10^5 Ω).

DMA Results of PVC Composite Films

Fig. 5 presents the DMA properties of various PVC composite films, showing the storage modulus (E', Fig. 5a) and loss factor (tanδ, Fig. 5b) with 4% plasticizers. The temperature corresponding to the tanδ peak is considered to be the glass transition temperature (T_g), and the shift of the tanδ peak to the left indicates a decrease in T_g.[46] The specific T_g values of PVC films containing different plasticizers are summarized in Table 1. DOP, a commonly used plasticizer, obviously reduces E' and T_g of PVC, suggesting a plasticizing effect but lower strength. By incorporating (P)ILs, the T_g values also decreased, whereas the E' values increased. In particular, the incorporation of 4% $PImC_6NTf_2$ increased E' to 2734.7 MPa, a 26.1% increase compared to that of

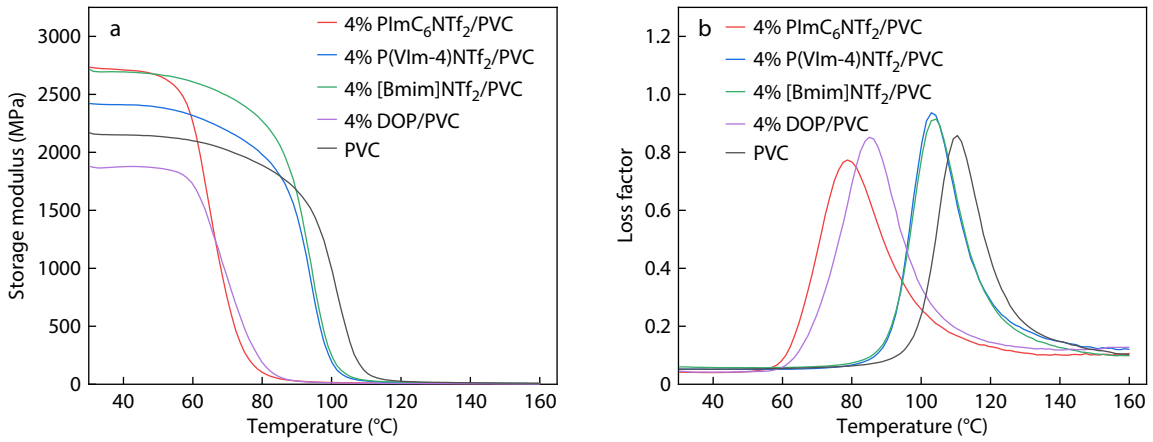

Fig. 5 DMA results of PVC composite films with 4% plasticizers: (a) storage modulus, (b) loss factor.

Table 1 T_g of PVC composite films.

Sample	T_g (°C)
PVC	110.34
4%DOP/PVC	85.36
4%[Bmim]NTf_2/PVC	103.87
4%P(VIm-4)NTf_2/PVC	103.04
4%PImC_6NTf_2/PVC	78.93

the neat PVC film. The PImC_6NTf_2/PVC composite films exhibited higher E' and lower T_g values than those of the other two (P)ILs and DOP-incorporated PVC films. These results suggest that PImC_6NTf_2 remarkably enhances the processability and mechanical properties of PVC.

Migration of Plasticizers in PVC Composite Films

The PVC composite films were immersed in ethanol and n-hexane at 50 °C for 1, 7, and 14 days to evaluate the migration resistance of the plasticizers.[47] As shown in Fig. 6, after 1 day in ethanol, the DOP/PVC film showed 2.0% weight loss, while no significant loss occurred in any (P) IL-containing film. With prolonged immersion, the (P) IL-containing PVC films gradually exhibited a weight loss. After 14 days, the weight losses of the DOP/PVC, [Bmim]NTf_2/PVC, PImC_6NTf_2/PVC, and P(VIm-4)NTf_2/PVC films reached 3.38%, 1.57%, 1.62%, and 1.65%, respectively. In *n*-hexane, the weight loss was more pronounced. After 1 day, the weight loss of the DOP/PVC film was as high as 3.30%, while the (P) IL-containing films still exhibited no weight loss. After 14 days, the weight losses of the DOP/PVC, [Bmim]NTf_2/PVC, PImC_6NTf_2/PVC, and P(VIm-4)NTf_2/PVC films reached 3.8%, 3.06%, 2.54%, and 2.85%, respectively. This study found that, compared to traditional DOP, (P)ILs demonstrate superior migration resistance in PVC. This is attributed to the stronger hydrogen bonding and electrostatic interactions with the PVC matrix. Additionally, the plasticizers exhibited faster migration rates and higher migration extents in *n*-hexane than in ethanol. This is due to the stronger swelling capacity of *n*-hexane on the PVC polymer network and its non-polar nature, which results in better compatibility with plasticizers.[48]

Tensile Properties of PVC Composite Films

Fig. 7 compares the effects of DOP and different (P)ILs on the

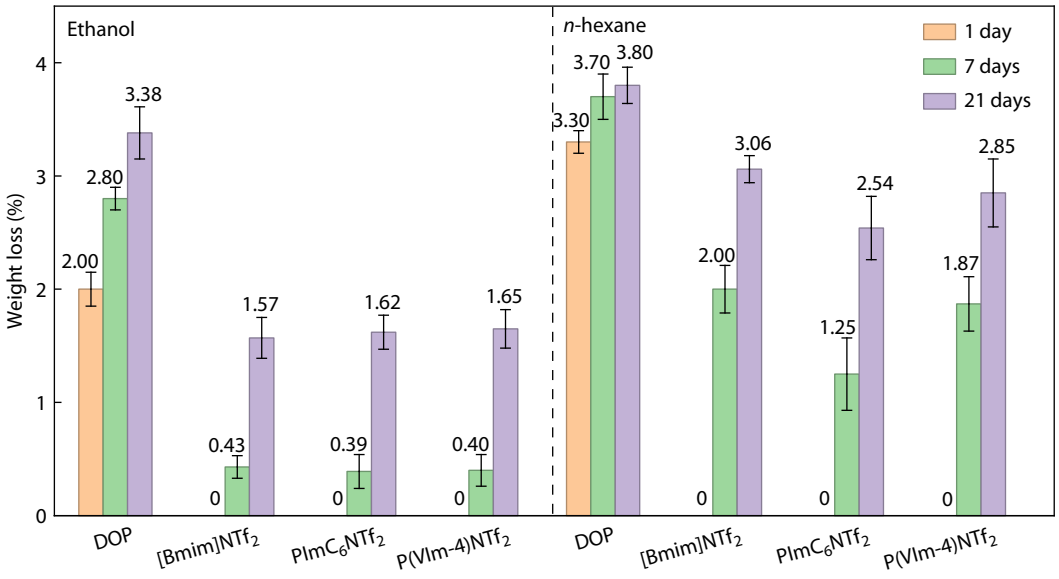

Fig. 6 The weight loss of PVC films containing 4 wt% of different plasticizers in absolute ethanol and *n*-hexane over time.

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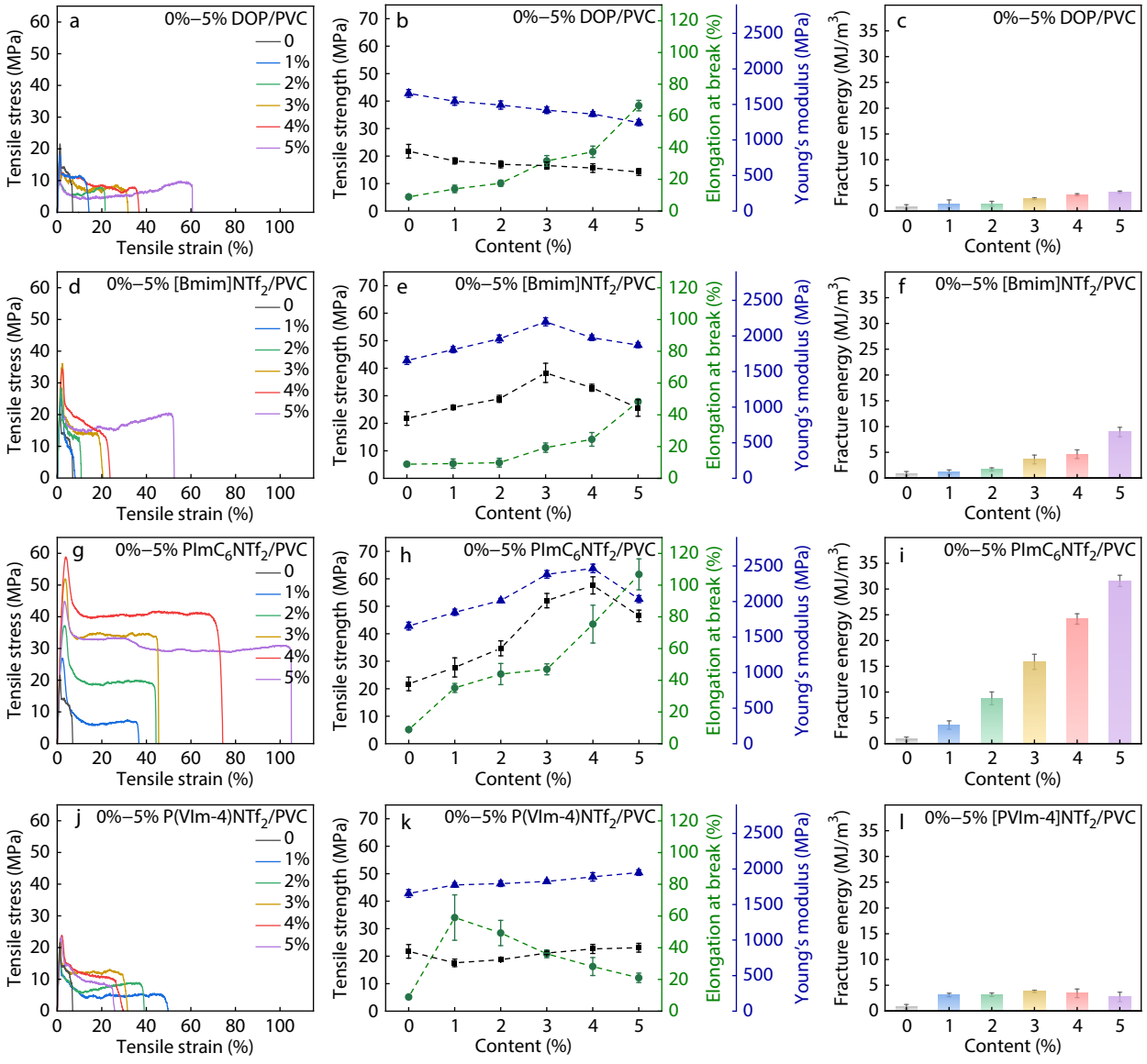

Fig. 7 Tensile properties of (a–c) DOP/PVC, (d–f) [Bmim]NTf_2/PVC, (g–i) $PImC_6NTf_2$/PVC, and (j–l) P(VIm-4)NTf_2/PVC composite films: (a, d, g, j) typical tensile stress-strain curves; (b, e, h, k) the tensile strength, elongation at break, and Young's modulus; (c, f, i, l) the fracture energy.

tensile properties of PVC composite films. For the neat PVC film, the elongation at break, tensile strength, and Young's modulus were only 8.9%, 21.8 MPa, and 1655 MPa, respectively (Figs. 7a and 7b). DOP significantly enhances the film toughness, but compromises its strength. As its content increased from 1% to 5%, the elongation at break increased from 13.9% to 66.5%, while the tensile strength and Young's modulus decrease from 18.2 MPa and 1544 MPa to 14.2 MPa and 1244 MPa, respectively.

In contrast, the three (P)ILs enhanced both toughness and strength, with $PImC_6NTf_2$ showing the best overall performance. For [Bmim]NTf_2, as its concentration increased, both the tensile strength and Young's modulus initially increased and then declined, whereas the elongation at break steadily increased (Figs. 7d and 7e). The highest tensile strength (38.3 MPa) and Young's modulus (2199 MPa) were observed with 3% [Bmim]NTf_2 incorporation, with the corresponding elongation at break reaching 19.3%. Most notably, $PImC_6NTf_2$ significantly improved both the toughness and strength. Compared with the neat PVC film, the elongation at break, tensile strength, and Young's modulus of 4%$PImC_6NTf_2$/PVC increased by 747.2%, 165.4%, and 49.1%, respectively (Figs. 7g and 7h). The fracture energy reaches 24.2 MJ/m^3, increasing by a factor of 27.4 (Fig. 7i). For 4%DOP/PVC, the elongation at break increased by 320.2%, whereas the tensile strength and Young's modulus decreased by 28.1% and 17.5%, respectively. For P(VIm-4)NTf_2/PVC, the elongation at break reaches its maximum value (59.0%) at 1% P(VIm-4)NTf_2 incorporation, then decreases at higher incorporations, while both tensile strength and Young's modulus improve gradually, from 17.6 MPa and 1776 MPa to 23.1 MPa and 1950 MPa, respectively (Figs. 7j and 7k). Overall, the fracture energies of the $PImC_6NTf_2$/PVC composite films were much higher than those of the other films (Figs. 7c, 7f, 7i and 7l), suggesting a significant increase in the toughness.

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Fig. 8 shows SEM images of the fracture surfaces of the PVC composite films after tensile testing. The neat PVC film exhibited a smooth fracture surface (Fig. 8a), indicative of brittle fracture, which is characteristic of PVC's high sensitivity of PVC to microcracks. Figs. 8(b)–8(e) present the fracture morphologies of the PVC composite films containing 4% DOP and different (P)ILs. These films displayed rougher and more irregular fracture surfaces, which are typical of ductile fractures. The transition from brittle to ductile fracture behavior was primarily attributed to the plasticization of DOP and (P)ILs. This resulted in a significant improvement in the flexibility and ductility of the PVC films, leading to increased elongation at break.[49]

The simultaneously improved strength and toughness are primarily attributed to hydrogen bonding and electrostatic interactions between the (P)ILs and PVC chains.[50,51] ATR-FTIR was used to characterize interactions in PVC composite films (Fig. S4 in ESI). In the [Bmim]NTf_2/PVC composite film, the C—F band shifted from 1200 cm^{-1} in bulk (P)ILs to 1193 cm^{-1} and the S—N—S band shifted from 1059 cm^{-1} to 1057 cm^{-1}. In $PImC_6NTf_2$/PVC, the C—F and S—N—S bands shifted to 1195 and 1056 cm^{-1}, respectively. For P(VIm-4)NTf_2/PVC, the S—N—S band shifted to 1195 cm^{-1} but the undetected S—N—S band shifted. The shifts are caused by van der Waals forces and dipole-dipole interactions between PVC and the (P)ILs, which contain a strong electronegative region.[52]

Fig. 9 illustrates the mechanism of DOP-and (P) IL-plasticized PVC. The intercalated DOP molecules formed very weak hydrogen bonds with PVC chains. Instead, they reduce the intermolecular forces between the PVC chains, thereby dissipating the concentrated stress during tensile deformation (Fig. 9a). In contrast, intercalated (P)ILs form hydrogen bonds as well as electrostatic forces, resulting in new cross-linking interactions. Specifically, [Bmim]NTf_2 molecules were distribut-

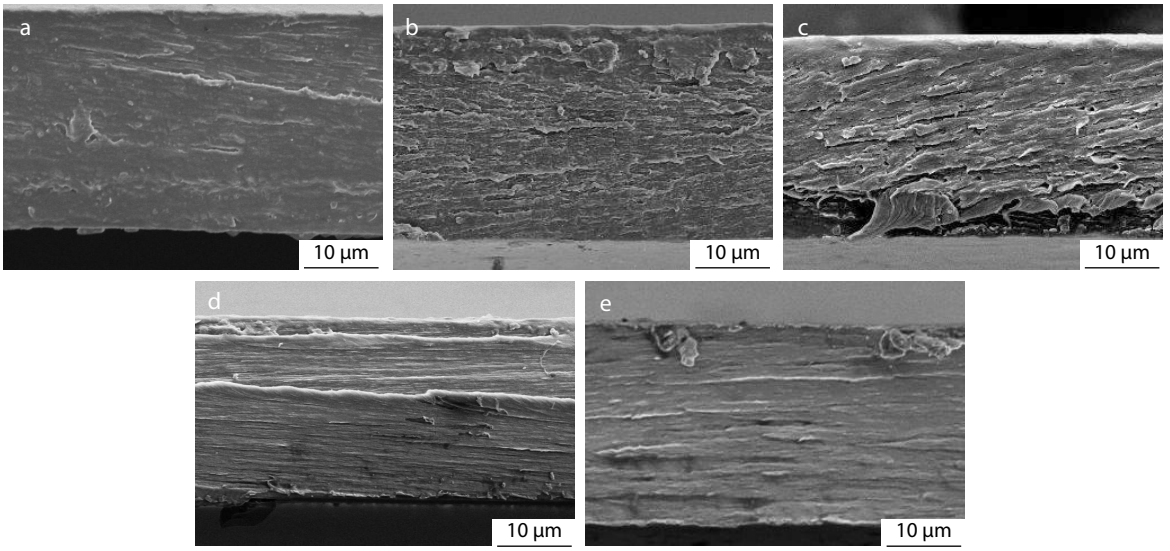
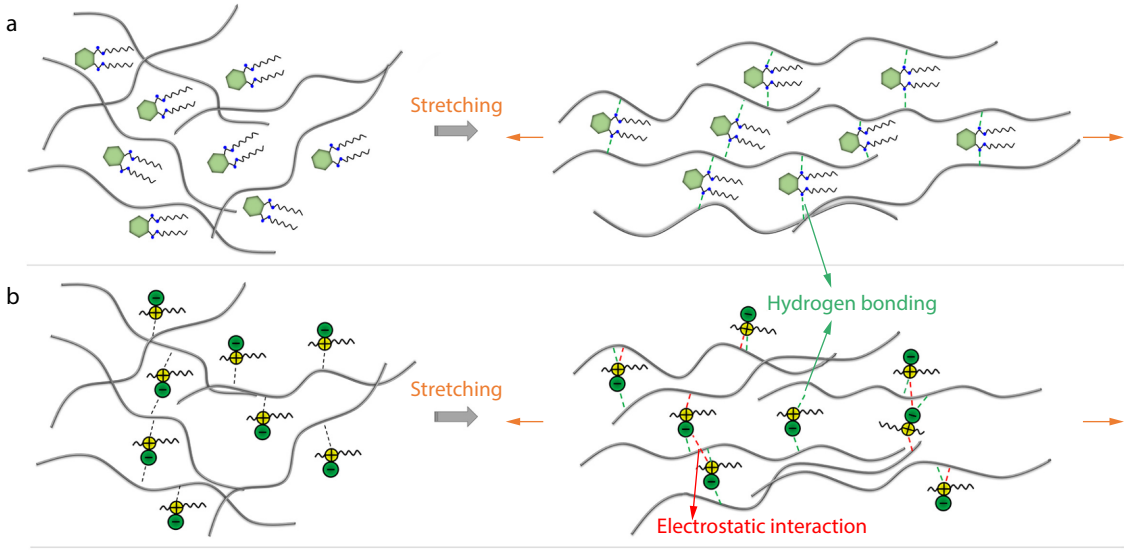


Fig. 8 SEM images of the fracture surfaces of (a) PVC, (b) 4%DOP/PVC, (c) 4%[Bmim]NTf_2/PVC, (d) 4%$PImC_6NTf_2$/PVC, and (e) 4%P(VIm-4)NTf_2/PVC films.



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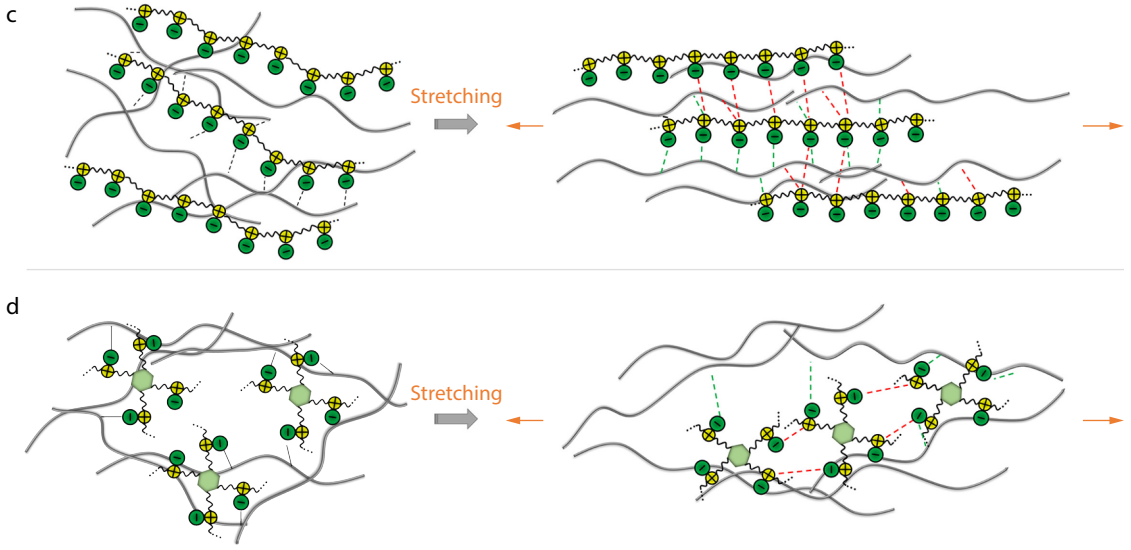

Fig. 9 Schematic diagram of the mechanism of DOP or ILs plasticizing PVC: (a) DOP, (b) [Bmim]NTf_2, (c) $PImC_6NTf_2$, and (d) P(VIm-4)NTf_2. Note that the hydrogen bonding between PVC chains themselves is not depicted.

ed around the PVC chains (Fig. 9b), whereas $PImC_6NTf_2$ molecules aligned more closely with the PVC chains to form stronger interactions (Fig. 9c). The $PImC_6NTf_2$ molecules weakened the intermolecular forces of PVC, but the cross-linking interactions between $PImC_6NTf_2$ and PVC chains caused the molecular chains to entangle.[53,54] The cross-linking interactions are dynamic, which not only promotes energy dissipation but also facilitates dynamic adhesion between molecules, thereby substantially improving the toughness and mechanical strength of PVC films.[55] The branched architecture of P(VIm-4)NTf_2 induced a pronounced steric hindrance effect. This effect impedes close-range interactions between the anionic centers and chlorine atoms on the PVC chains, resulting in weak interfacial binding (Fig. 9d). As the concentration increased, such steric hindrance further suppressed the tight association with PVC chains, thereby diminishing the plasticizing efficiency.[56]

Fig. 10 summarizes the tensile properties of PVC composites plasticized with different plasticizers in the references and (P)ILs in this study. Most conventional plasticizers exhibit a toughening effect but negatively affect the mechanical strength to some extent. These plasticizers increase the free volume by reducing the intermolecular forces between polymer chains. We also found that certain plasticizers, namely epoxidized 1,6-hexanediol oleate (EHD) and dioctyl adipate (DOA), simultaneously exhibited reinforcing and toughening effects. They appropriately weakened the interactions between the PVC molecular chains, thereby enhancing their toughness. Compared to conventional plasticizers, EHD and DOA have straight-chain alkyl and carbonyl groups, respectively. They can form stronger interactions with PVC molecular chains *via* hydrogen bonding and electrostatic interactions, further enhancing the mechanical properties of the material.[52] The addition of 1-butyl-3-methylimidazolium hexafluorophosphate ([Bmim]NTf_2) and 1-hexyl-3-methylimidazolium hexafluorophosphate ([Hmim]NTf_2) resulted in extremely high plasticizing effects on PVC. However, owing to the excessively high plasticizer content, the strength decreased substantially. By comparing the effects of different plasticizers, including the (P)ILs employed in this study, we conclude that appropriately strong interactions between plasticizers and PVC can concurrently improve both toughness and strength. Moreover, significant improvements can be achieved by carefully regulating the molecular structure of (P)ILs, especially linear PILs.

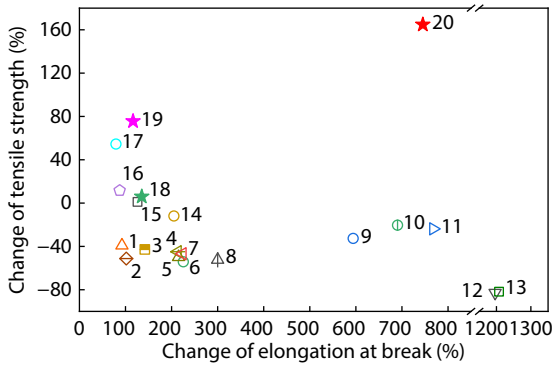

Fig. 10 Comparison of various plasticizers' impact on the tensile properties of PVC. Specifically, (1) phosphorous based epoxidized simarouba glauca seed oil (25%)[57], (2) epoxy 1,6-hexanediol oleate and dioctyl adipate (26.7% and 6.6%)[58], (3) epoxidized linoleic acid cardanol ester (28.6%)[59], (4) epoxidized castor oil polyol ester (28.6%)[60], (5) ethylene glycol oleate (20%)[47], (6) glucose oleate (20%)[61], (7) *N*-((tetrahydrofuran-2-yl)methyl)undecane-2-amine (22%)[62], (8) di(2-ethylhexyl)phthalate (22%)[62], (9) branched lactic acid ester (28.6%)[63], (10) acetyl tributyl citrate (28.6%)[63], (11) hyperbranched cardanol epoxy polyglycerol (22.2%)[64], (12) 1-butyl-3-methylimidazolium hexafluorophosphate ([Bmim]NTf_2, 40%)[49], (13) 1-hexyl-3-methylimidazolium hexafluorophosphate ([Hmim]NTf_2, 40%)[49], (14) dioctyl phthalate and epoxidized cassia fistula seed oil (28.1% and 9.38%)[65], (15) trioctyl propane-1,2,3-tricarboxylate (5%)[66], (16) epoxidized 1,6-hexanediol oleate (33.3%)[67], (17) dioctyl adipate (33.3%)[58] and (18–20) in this work.

Tribological Properties of PVC Composite Films

Fig. 11 shows the wear volume and friction coefficient of the

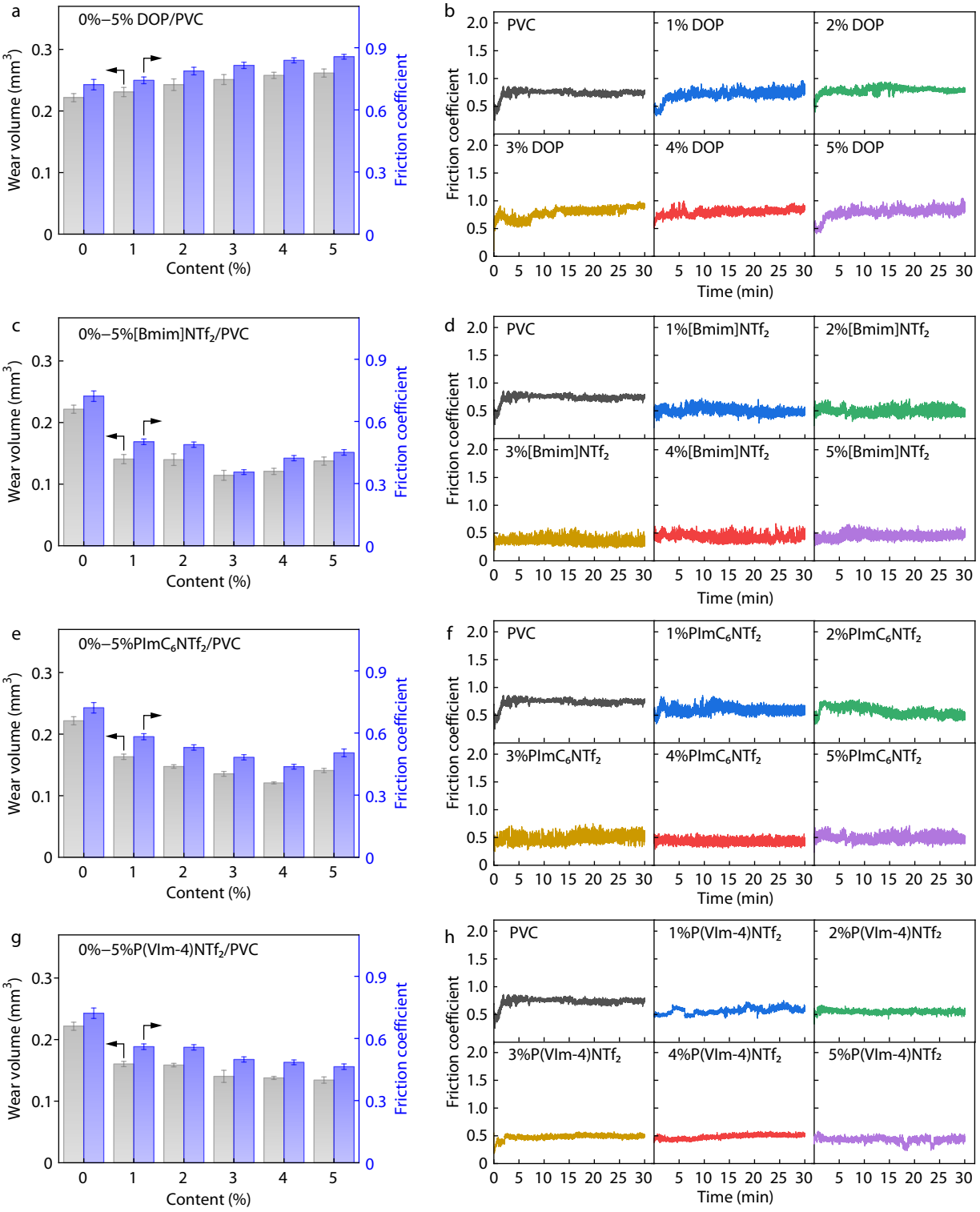

Fig. 11 (a, c, e, g) Wear volume, friction coefficient, (b ,d ,f ,h) online friction coefficient of PVC composite films: (a, b) 0%–0.5% DOP/PVC, (c, d) 0%–0.5% [Bmim]NTf_2/PVC, (e, f) 0%–0.5% $PImC_6NTf_2$/PVC, (g, h) 0%–0.5% P(VIm-4)NTf_2/PVC.

PVC composite films. For the neat PVC film, the wear volume was 0.222 mm^3 and the friction coefficient was 0.722. During testing, the friction coefficient gradually increased and then stabilized at a constant value (Fig. 11b). For the DOP/PVC composite films, both the wear volume and friction coefficient increased as the DOP content increased (Fig. 11a). The overall friction coefficient followed a trend similar to that of the neat PVC (Fig. 11b). The inserted DOP molecules weaken the intermolecular forces between the PVC chains, reducing the energy required for chain segment movement, which in turn leads to a

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decrease in the wear resistance and friction-reducing performance.

In contrast, PVC films containing (P)ILs exhibited significant improvements in their tribological properties. For the [Bmim]NTf_2/PVC films, as the [Bmim]NTf_2 content increased, both the wear volume and friction coefficient initially decreased and then increased, with optimal values observed at a 3% content (Figs. 11c and 11d). At this concentration, the wear volume and friction coefficient were reduced by 48.6% and 50.8%, respectively, compared to those of the neat PVC film. For the $PImC_6NTf_2$/PVC films, the wear volume and friction coefficient initially decreased and then increased with increasing $PImC_6NTf_2$ content (Figs. 11e and 11f). At the optimal content of 4%, the wear volume and friction coefficient were reduced by 45.5% and 39.5%, respectively, compared to those of the neat PVC film. Furthermore, as the P(VIm-4)NTf_2 content increased, both the wear volume and friction coefficient decreased steadily (Figs. 11g and 11h). The significant improvements in tribological properties can be primarily attributed to the enhanced strength of the composite films and excellent lubricating properties of the ILs.[68] During sliding, localized high temperatures and pressures are generated, causing partial damage or extrusion of the PVC film. This enabled the PILs within the PVC film to continuously migrate from the matrix interior to the friction interface. This achieves a dynamic equilibrium of the lubricating film and a certain degree of "self-healing" capability, thereby maintaining a long-term stable low-friction state.[69]

CONCLUSIONS

In this study, three (P)ILs with different structures (monomeric [Bmim]NTf_2, linearly polymerized $PImC_6NTf_2$, and branched polymerized P(VIm-4)NTf_2) were used as plasticizers to enhance the processing and mechanical properties of PVC. The DMA results demonstrate a decrease in the T_g of the PVC composite films containing (P)ILs, indicating an improvement in the processing performance. Mechanical property evaluations revealed that the PVC composite films containing (P)ILs exhibited significantly higher elongation at break and tensile strength than neat PVC. Notably, the elongation at break, tensile strength, and Young's modulus of the 4%$PImC_6NTf_2$/PVC film increased by 747.2%, 165.4%, and 49.1%, respectively, compared to those of the neat PVC film. In contrast, the elongation at break of the 4%DOP/PVC film increases by 320.2%, and the tensile strength and Young's modulus decreases by 28.1% and 17.5%. Furthermore, the incorporation of (P)ILs led to substantial improvements in the thermal stability, hydrophobicity, and wear resistance. This study presents a new approach for the development of high-performance PVC materials and opens up exciting possibilities for the use of (P)ILs in the plastics industry.

Conflict of Interests

The authors declare no interest conflict.

Electronic Supplementary Information

Electronic supplementary information (ESI) is available free of charge in the online version of this article at http://doi.org/10.1007/s10118-026-3618-5.

Data Availability Statement

The data supporting the findings of this study are available from the corresponding authors upon reasonable request.

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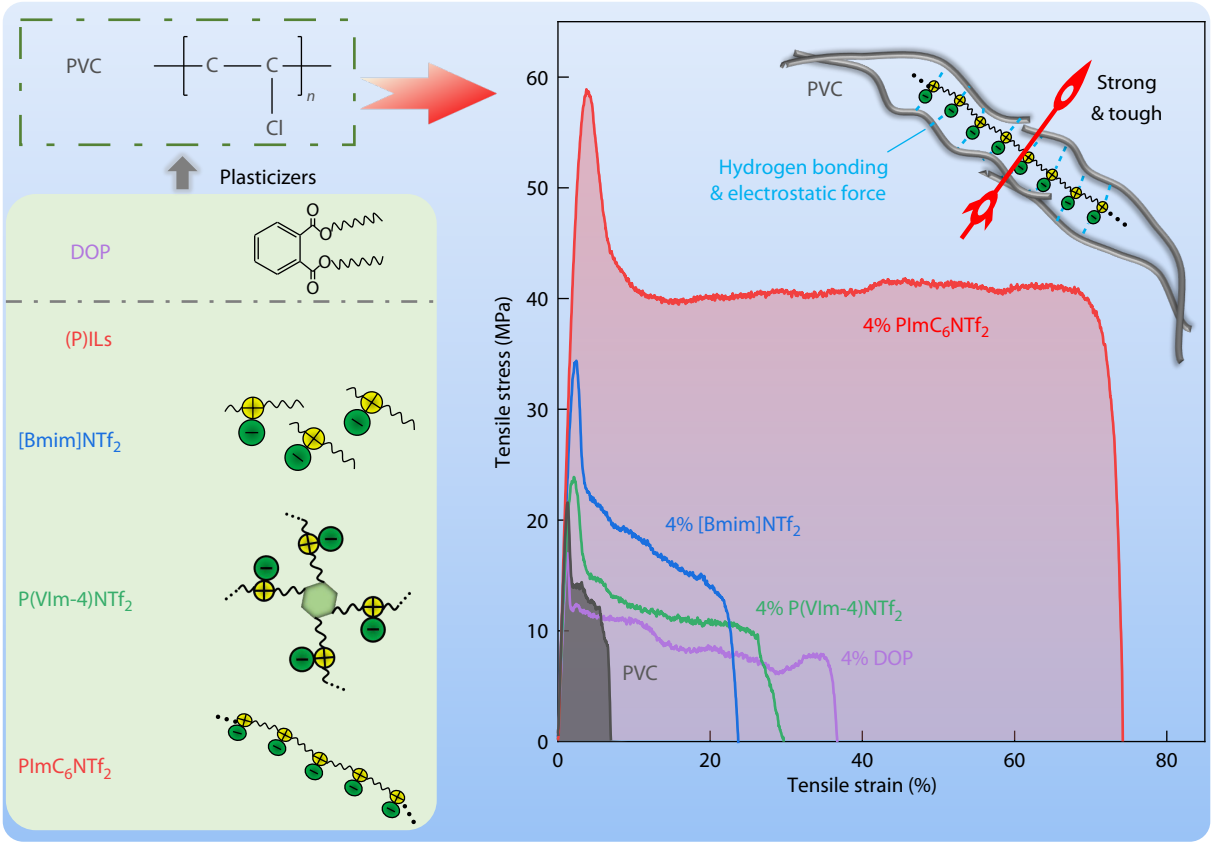
Graphical Abstract

Plasticizing and Strengthening Poly(vinyl chloride) with Structurally Different Ionic Liquids

Xiang Gao, Ji-Shuo Ye, Zhi-Ji Liu, Jian Wu, and Ning-Zhong Bao

Nanjing Tech University; Zhejiang University; Hangzhou City University

This work investigates the reinforcing effects of ionic liquid monomer, linear and branched poly(ionic liquids) on poly(vinyl chloride) (PVC). Linear poly(ionic liquids) exhibit the best strength and toughness, which are significantly superior to dioctyl phthalate (DOP).



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