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Transparent and heat-insulation plasticized polyvinyl chloride (PVC) thin film with solar spectrally selective property

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ABSTRACT

In this work, we present the development and assessment of transparent and heat-insulation thin films that represent a new class of polyvinyl chloride (PVC) polymers. The addition of ultraviolet (UV) absorber, yellow pigment and indium tin oxide (ITO) or antimony tin oxide (ATO) showed significant improvement upon shielding effect against UV light, blue light and near-infrared (NIR) light, respectively. Particularly, environmentally friendly polyester plasticizer was applied. The optical and actual heat insulation properties were investigated by an Ultraviolet–Visible–Near infrared (UV–vis–NIR) spectrometer and a self-designed device, respectively. It was found that low transmittance (21.09% and 24.46% respectively) in NIR region (800–2600 nm) can be achieved with the addition of 2.0 phr ITO or ATO particles, and a decrease of 3 °C or 5 °C can also be observed in temperature test. Meanwhile, the thin films possessed a low UV transmittance performance (200–400 nm), accompanied with low transmittance in blue light range (400–500 nm) while maintained a high light transmittance in visible light range (400–800 nm). The shielding effectiveness provided by the materials fabricated in this study offer specificity and potency in next-generation transparent and heat-insulation thin film, with applications in protection of valuable antique, glasses, display screen and windows of automobiles.

1. Introduction

Sunlight is essential to the circulation of the living system as the main energy source. Sunlight mainly consists of three major parts: ultraviolet (UV) light, visible (Vis) light and near infrared (NIR) light. The lethal germicidal effect of UV light on bacteria has been used extensively as a high-efficiency way of sterilization [1]. Blue light, a high-energy light source in visible wavelength, is a clinically effective phototherapy in bacterial destruction [2] and plays a significant role in maintaining some groups of phytoplankton [3]. Additionally, NIR light has been used extensively in the treatment of ischemic and hypoxic wounds as a well-accepted therapeutic tool [4]. However, aforementioned lights are harmful in particular situations. The energy of UV light source is strongly centralized due to its short wavelength. Overexposure to UV irradiation will induce inflammation, sunburn and immunologic changes of skin [5,6]. UV exposure can also cause the degradation of encapsulant materials, resulting in the loss in the transparency and service life [7]. High levels of exposure to blue light may lead to ocular damage and are associated with the development of age-related macular degeneration [8]. Moreover, thermal effect of NIR may accumulate unexpected heat [9], and increase energy consumption targeted to cool inner temperature. Thus, materials with excellent optical and heat-insulation properties have great potential applications, such as glass film for building or automotive, and protective films for color printing or culture relics.

To address these problems, current researches have predominately focused on engineering transparent thin films with optical properties [10–12], yet most of them concentrate on conductivity. Indeed, the addition of inorganic functional particles into polymer matrix has promoted more satisfying properties [13–15]. Hence, some additives have been developed to block UV irradiation, blue light or NIR rays to prepare optical thin films, including ZnO [5], fluorine doped tin oxide (FTO) [16], indium tin oxide (ITO) [11,13], antimony tin oxide (ATO) [14], and cesium tungsten bronze ($Cs_{0.33}WO_3$) [17], etc. ITO and ATO are widely known as n-type oxide semiconductors, which are also good candidates for conductive fillers, transparent electrodes, solar cells and display devices because of their low resistivity and high transmittance in the visible light range [18]. ITO and ATO are known to be opaque in the NIR wavelength [19,20], because of the free carrier absorption within the conduction band [21,22]. Recently, polymer matrices have been used to produce transparent thin films in many fields, such as food packing [18], coke hoses [23], clothing fibers [24], and screen protection [25], etc. Several types of polymer matrices, like polyvinyl chloride (PVC),

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polyethylene terephthalate (PET) [18,26] and ethylene-vinyl acetate copolymer (EVA) [27], have been used in the transparent thin film industry for a long time. Around a specific temperature of 127 °C and 69 °C respectively [24,27], PET and EVA would crystallize and became opaque. PET and EVA are abandoned in this study due to their crystallization behavior under a specific temperature [24,27], whereas PVC exhibits no crystallization absolutely. Besides being durable, stable, strong and flame resistant [28,29], it can also be mixed with various additives to yield other necessary properties for more applications [30], such as toys, wall covering and flooring. Specifically, plasticizers are used widely to manufacture flexible PVC [30] thin films. However, toxic phthalate plasticizers have been used widely, which damage the environment and threat human health. In our consideration, environmentally friendly polyester plasticizer was applied and plasticized PVC was chosen to be the polymer matrix owing to its considerable transparent and excellent mechanical properties [28].

However, literatures published almost focused on shielding only one part of the harmful lights [5,6,8,12,20], or studied NIR-shielding property correlated to conductivity narrowly [12,16]. The optical and actual heat-insulation properties of transparent materials have barely been studied together. Therefore, it is of great interest to create transparent and heat-insulation materials with property of shielding all unexpected lights.

Traditionally, depositing upon a substrate is a proverbial way to fabricate optical thin films. The thin films are usually deposited by thermal evaporation method [31,32], ion beam sputtering [18], magnetron sputtering [12], and laser ablation [32]. Nevertheless, melt blending method is another efficient way to fabricate optical thin films [33]. In this study, melt blending method was applied. ITO or ATO particles can be dispersed into the polymer substrate mechanically by fluid shear force in melting process.

In this study, plasticized PVC was used for its considerable transparent and perfect mechanical properties and environment friendly polyester plasticizers were used for green concept particularly. UV absorber and yellow pigment were applied to block UV irradiation and blue light, respectively. ITO or ATO particles were also introduced to shield NIR light. Thus, plasticized PVC/ITO and plasticized PVC/ATO thin films showed improved optical and heat-insulation properties while remaining highly transparent. Herein, the influence of UV absorber, yellow pigment and different content of ITO or ATO on transmittance and mechanical properties were systematically investigated. Furthermore, the heat-insulation property was also evaluated by a self-designed device.

2. Experimental

2.1. Materials and sample preparation

PVC resin (Type: S-1000) was purchased from China Petrochemical Co. Ltd., Qilu Branch. ITO and ATO were supplied by Nanjing Haitai nano-materials Co. Ltd., China. Polyester plasticizer (2230) was from Dainippon Ink and Chemicals Inc., Japan. Organotin stabilizer (T-137) was provided by Arkema Beijing Chemical Co. Ltd., China. Liquid paraffin used as lubricant was produced by Jinling Petrochemical Co. Ltd., China. UV absorber (UV326) was obtained from Ciba Specialty Chemicals Co. Ltd., Switzerland. Yellow pigment (PY14) was supplied by Nanjing Huage Electronics and Automobile Plastic Industry Co. Ltd., China.

The synthesis formulation is defined in Table 1. The amount of additives is calculated by per hundred ratio (phr) to the mass of PVC resin. Plasticized PVC (100 phr PVC, 65 phr polyester plasticizer, 1.5 phr organotin, 0.6 phr liquid paraffin, 0.5 phr UV absorber (UV326) and 0.01 phr yellow pigment) was used as a control and was melt-blended with different content of ITO or ATO particles for 5 min in a two-roll mill at 150 °C. The mixtures were subsequently compressed into 0.3 mm and 1 mm thickness thin films at 160 °C under a pressure of 10 MPa before further characterizations. 0.3 mm thickness thin films were used in transmittance spectra, light transmittance and haze measurement, while 1 mm thickness thin films were used for mechanical and heat-insulation properties test.

Table 1
Synthesis formulation of specimens blended with different amount of additives.

PVC	Polyester plasticizer	Organotin	Liquid paraffin	UV absorber	Yellow pigment	ITO	ATO
100	65	1.5	0.6	0.5	0.01	–	–
100	65	1.5	0.6	0.5	0.01	0.5	–
100	65	1.5	0.6	0.5	0.01	1.0	–
100	65	1.5	0.6	0.5	0.01	1.5	–
100	65	1.5	0.6	0.5	0.01	2.0	–
100	65	1.5	0.6	0.5	0.01	–	0.5
100	65	1.5	0.6	0.5	0.01	–	1.0
100	65	1.5	0.6	0.5	0.01	–	1.5
100	65	1.5	0.6	0.5	0.01	–	2.0

2.2. Characterization

Light transmittance and haze were measured by an automatic transmittance haze meter (WGT-S, Shanghai instrument physical optics instrument Co. LTD., China) according to ASTM D 1003 standard. Ultraviolet–Visible–Near infrared (UV–vis–NIR) spectrometer (UV3101PC, Shimadzu Co., Japan) was used to obtain transmittance spectra, ranging from 200 to 2600 nm. Transmittance (T) was proposed to evaluate the transmittance in different regions, which can be calculated by:

$$T = \frac{\int_{\lambda_1}^{\lambda_2} T(\lambda) d\lambda}{\lambda_2 - \lambda_1} \quad (1)$$

where $T(\lambda)$ is the transmittance value at the wavelength (λ); λ_1 and λ_2 are the minimum and maximum wavelength values of a solar region, respectively.

Actual heat-insulation property was assessed using a previous protocol [34], by a self-designed device [35]. The self-designed device is a high heat-insulation polymeric cylinder with a thermometer. The samples were all mounted on the glass surface of the device when the temperature test was conducted, on a sunny day. Another device without samples is also needed at the same time and the same place as a control. The internal temperatures are read during a certain time interval.

The tensile and tearing properties of the thin films were investigated by a universal testing machine (CMT 5254, Shenzhen SANS Testing Machine Co., Ltd., China) at a testing rate of 200 mm/min, according to GB/T 13022-1991 and GB/T 529-91, respectively.

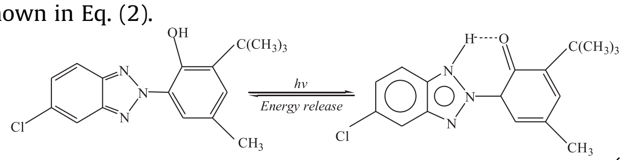
3. Results and discussion

3.1. Light transmittance and haze

Table 2 demonstrates the light transmittance and haze of the mixtures. The light transmittance of the thin films is negatively correlated with increasing ITO or ATO content, and the increase in haze is accompanied simultaneously. With the addition of 0.5 phr ITO content, the light transmittance and haze are 85.47% and 6.57%, respectively. By adding 2.0 phr ITO content, the light transmittance declines to 76.34%, while the haze goes up to 16.08%. With regard to the plasticized PVC/ATO thin films, the addition of 2.0 phr ATO into the matrix decreases the light transmittance to 66.23%, and increases the haze to 62.83%. On the other hand, the presence of ITO particles allows the materials with higher light transmittance and lower haze than ATO. Overall, although ITO and ATO particles perform a refutation to visible light transmittance, thin films can maintain a receivable level of transparency because of the homogenous hybrid construction of the blends.

3.2. Transmittance measured by UV–vis–NIR

The spectral transmittance and the relatively regulated transmittance of specimens are illustrated in Fig. 1 and Table 3, respectively. In the UV range, the samples all show an extremely abrupt increase around the wavelength at 400 nm, indicating that the thin films mixed with UV absorber (UV326) show excellent shielding efficiency of UV light blocking up to 400 nm. UV326 possesses a significant property of a mechanism for the rapid dissipation of the absorbed UV radiation via some suitable intramolecular rearrangement [36]. For UV326, the benzotriazole-type of UV absorber, the mechanism of excited-state deactivation is because of an excited-state intramolecular proton transfer as shown in Eq. (2).



(2)

UV326 contains an intramolecular hydrogen in the ground state, and can move up to the excited-state when UV radiation is preferentially absorbed. The energy of UV radiation absorbed will be dissipated in the form of heat in an ultrafast time. Surprisingly, UV326 exhibits amazing UV shielding ability but maintains a high optical transmittance feature of mixtures in the visible light wavelength. Meanwhile, a slight decrease of transmittance in blue light region (400–500 nm) is observed in solar spectra. The little light absorption ranges from 400 to 500 nm is ascribed to the law of complementary color, which result in the blue light shielding ability of yellow pigment [8]. Additionally, the high NIR-shielding property combined with high transmittance in visible wavelength is achieved due to the addition of ITO or ATO particles. As 0.5 phr ITO or ATO content are added, the transmittance in NIR region of samples are 43.19% or 65.96%, respectively, while retaining a high visible transmittance of 77.84% or 66.69%, respectively. Curves in Fig. 1 clearly indicate that the NIR-shielding property is enhanced by increasing ITO or ATO content with the sacrifice of visible spectral transmittance. While the ITO content increases from 0 phr to 2.0 phr, the transmittance in NIR region is successfully blocked from 81.21% to 21.09%, while the visible transmittance is decreased from 81.11% to 65.57%. In terms of the plasticized PVC/ATO thin films, a drastic reduction close to 70% of the transmittance in NIR region is achieved with 2.0 phr ATO content, compared to control.

Table 2
Light transmittance and haze of all specimens added with different amount of additives.

Samples	Light transmittance (%)	Haze (%)
Control	88.23 ± 0.19	3.74 ± 0.17
0.5 phr ITO	85.47 ± 0.09	6.57 ± 0.22
1.0 phr ITO	83.83 ± 0.21	6.99 ± 0.12
1.5 phr ITO	79.43 ± 0.24	11.65 ± 0.04
2.0 phr ITO	76.34 ± 0.54	16.08 ± 0.51
0.5 phr ATO	84.73 ± 0.25	19.22 ± 0.51
1.0 phr ATO	76.60 ± 0.22	41.26 ± 0.27
1.5 phr ATO	74.40 ± 0.43	48.54 ± 1.68
2.0 phr ATO	66.23 ± 0.29	62.83 ± 0.31

However, the visible light transmittance is sacrificed, decreasing to 24.73%, far lower than that of plasticized PVC/ITO thin films. As we all know, one oxygen vacancy owns two free electrons. According to the band theory, the electrons of ITO or ATO particles will absorb UV light and move up from ground state to excited state to form electronic-holes once sunlight illuminates the materials [22]. When electrons move back, the other wavelengths of photons will scatter or absorb. Because photon energy is less than the forbidden band width of ITO or ATO particles, the particles will no longer produce intrinsic absorption. Therefore, ITO or ATO particles will block NIR light once sunlight irradiates.

ITO or ATO particles are opaque in NIR region because of the free carrier absorption within the conduction band [21], according to classical Drude theory [20,37]:

$$\omega = \sqrt{\frac{e^2 N}{\varepsilon_0 m}} \tag{3}$$

where e is the charge of electron, N is the charge carrier concentration for the Plasmon frequency, ε_0 is the permittivity of the free space and m is the effective mass of free electron. The plasma frequency (ω) presents the infrared crossover from high transmittance to high reflectivity.

According to Eq. (3), the plasma wavelength (λ_p) follows the equation [38]:

$$\lambda_P \propto \sqrt{\frac{m}{N}} \tag{4}$$

where N means the charge carrier concentration and m represents the effective mass of free electron similarly.

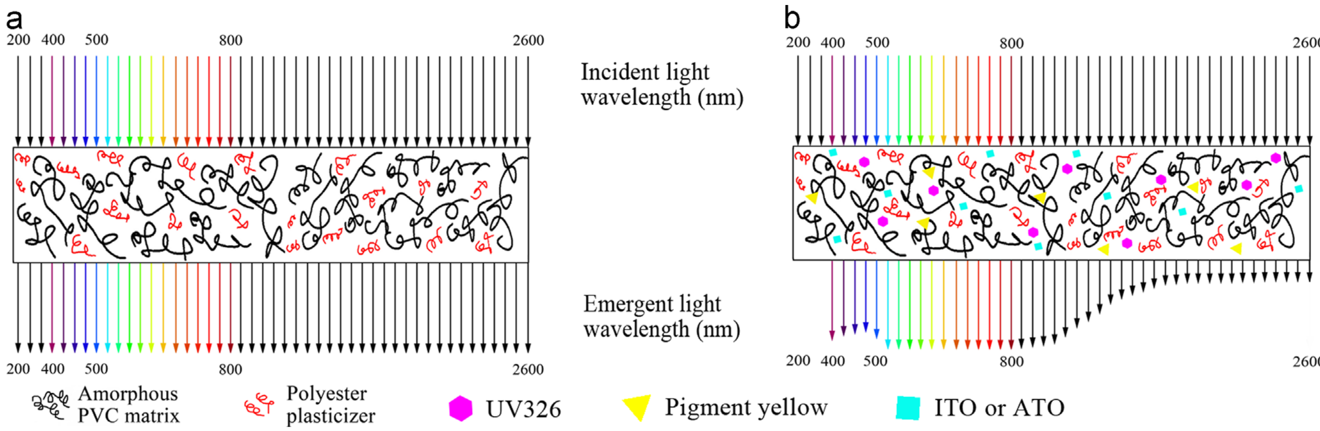
Particularly, the NIR shielding efficiency is determined by the free carrier concentration within the samples. With increasing content of ITO or ATO, the charge carriers increase as more number of free electrons is available and increased mobility of electrons happen [37,39]. As shown in Eq. (4), the plasma edge moves to shorter wavelengths with increased carrier concentration [38]. After plasma edge, transmittance would be low and reflectance would be improved. As a result, a more gradual decrease in the transmittance and increase in the reflectance can be observed in Fig. 1 with increasing ITO or ATO content. Hence, the NIR blocking ability improved with the increasing ITO or ATO content.

Scheme 1 describes the solar spectrally selective property of PVC thin film. Without the addition of UV326, yellow pigment and ITO or ATO particles, the thin film does not possess solar spectrally selective property, shown in Scheme 1(a). Ultraviolet absorber is used widely to filter UV light and dissipates the energy in the form of heat. As shown in Scheme 1(b), UV326 shields the UV light effectively at a range up to 400 nm. According to the theory of

Table 3
Transmittance of specimens added with different amount of additives.

Samples	Transmittance/%			
	T_{UV}	T_B	T_{Vis}	T_{NIR}
Control	0.54	62.43	81.11	81.21
0.5 phr ITO	0.70	58.64	77.84	43.19
1.0 phr ITO	0.47	52.20	74.42	30.68
1.5 phr ITO	0.28	43.15	69.69	24.51
2.0 phr ITO	0.19	36.34	65.57	21.09
0.5 phr ATO	0.63	51.43	66.69	65.96
1.0 phr ATO	0.16	28.04	44.10	42.38
1.5 phr ATO	0.70	25.10	38.32	37.57
2.0 phr ATO	0.08	13.93	24.73	24.46

T_{UV}=Transmittance in ultraviolet light region (200–400 nm); T_B=Transmittance in blue light region (400–500 nm); T_{Vis}=Transmittance in visible light region (400–800 nm); T_{NIR}=Transmittance in near infrared light region (800–2600 nm).



Scheme 1. Schematic diagram of ultraviolet shielding, blue light filtering and near infrared light blocking properties of plasticized PVC films: (a) without UV absorber (UV326), pigment yellow and ITO or ATO particles; and (b) added with UV absorber (UV326), pigment yellow and ITO or ATO particles.(For interpretation of the references to color in this figure, the reader is referred to the web version of this article).

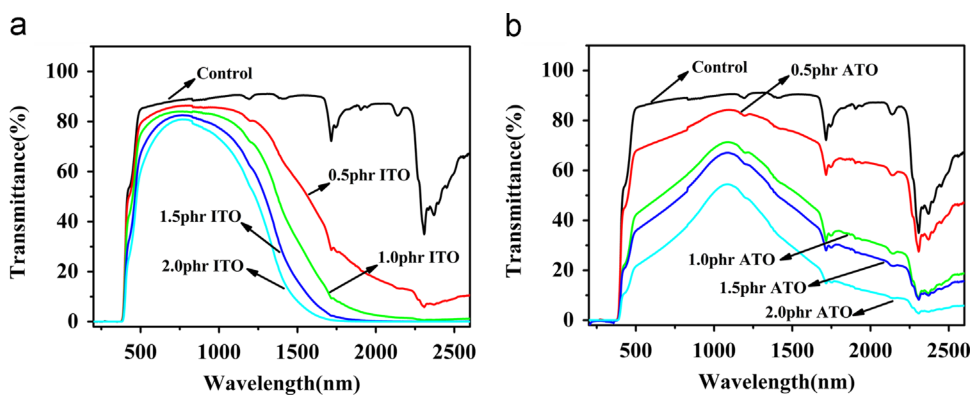


Fig. 1. Transmittance curves of specimens added with different amount of additives: (a) ITO; (b) ATO.

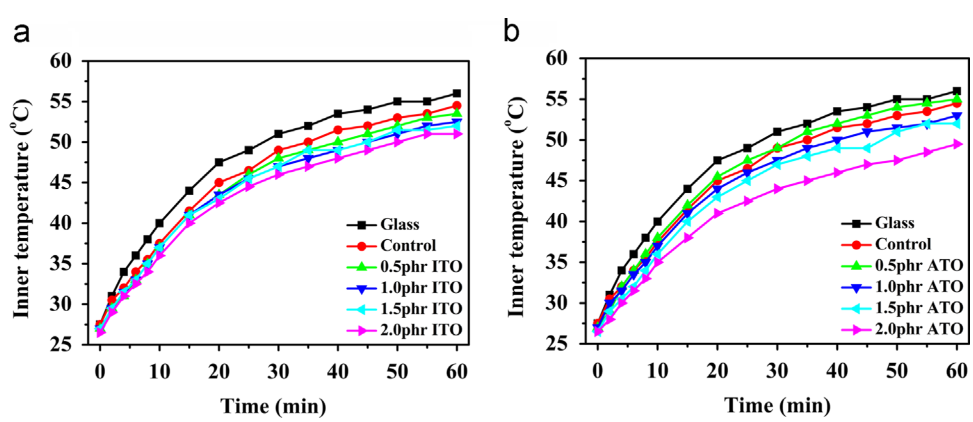


Fig. 2. Inner temperature changes of the devices within a period of time: (a) ITO; (b) ATO. This temperature test is conducted from 10:00 to 11:00 am on 23rd July 2014. The real-time temperature of the environment is 30 °C, 32°4′37″N, 118°46′19″E, in city Nanjing, China.

complementary colors, the yellow and the blue, the light of two complementary colors, can give a mutual blocking effect. Hence, appropriate amount of yellow pigment can be used as blue light absorber to prevent high-energy visible light. ITO or ATO particles can be functionalized as NIR shielding particles because of free carrier absorption. The resultant effect of UV326, yellow pigment and ITO or ATO particles is displayed in Scheme 1(b). The thin film possesses UV light shielding, filtration of blue light and NIR blocking, combined with high transparency in visible light region.

3.3. Temperature test

The inner temperature changes of the devices within a period of time are summarized in Fig. 2. The slope of initial temperature-

Table 4
Mechanical properties of specimens added with different amount additives.

Samples	Tensile strength (MPa)	Modulus at 100% (MPa)	Elongation at break (%)	Tearing property (kN/m)
Control	13.12 ± 1.67	7.14 ± 0.48	223 ± 30	46.59 ± 2.09
0.5 phr ITO	15.53 ± 0.30	12.06 ± 0.34	159 ± 6	54.81 ± 1.90
1.0 phr ITO	16.47 ± 0.66	11.26 ± 0.39	190 ± 17	51.15 ± 1.96
1.5 phr ITO	15.28 ± 0.70	11.96 ± 0.08	153 ± 13	53.02 ± 3.31
2.0 phr ITO	16.12 ± 2.17	11.03 ± 1.16	192 ± 18	51.46 ± 7.20
0.5 phr ATO	14.47 ± 0.72	5.87 ± 0.23	359 ± 9	49.00 ± 2.21
1.0 phr ATO	14.43 ± 0.94	5.45 ± 0.07	370 ± 35	51.17 ± 4.93
1.5 phr ATO	14.49 ± 1.02	5.96 ± 0.18	351 ± 23	52.22 ± 0.42
2.0 phr ATO	14.55 ± 0.38	6.22 ± 0.18	352 ± 20	51.25 ± 1.11

rise stage of specimens added with ITO or ATO particles is flatter than that of specimens without functional particles. Subsequently, the inner temperature increases up to an equilibrium point. Therefore, the addition of ITO or ATO particles can improve the heat insulation characteristic of substrate. Besides, the cooling ability is enhanced with the increasing ITO or ATO content. The high NIR-shielding ability prevents the heat conduction from upper thin film to materials underlying, lowering the inner temperature and protecting materials from heat damage caused by NIR irradiation.

3.4. Mechanical properties

Mechanical properties of the thin films with different concentration of ITO or ATO were measured to reveal the efficiency of mechanical improvement by ITO or ATO particles. Tensile and tearing properties are shown in Table 4. Plasticized PVC is reinforced by the addition of ITO or ATO particles to some extent. Though the elongation at break has decreased, an improvement of tensile strength and modulus at 100% of the thin films is attained when ITO are added into plasticized PVC (control). The reduction of elongation at break should be due to the aggregation of the ITO particles happens. The poor dispersion of ITO particles in the plasticized PVC matrix causes the reduction of the extensibility of the thin films. With regard to the thin films added with ATO particles, the tensile strength and elongation at break have been promoted, while the modulus at 100% has decreased. Particularly, the large improvement of elongation at break is mainly attributed to the interface effect of the ATO particles. Due to the molecular chains are absorbed firmly by the surface of ATO particles, the physical cross-linking structure is formed to improve the elongation at break values of the thin films [40]. As to the values of modulus at 100%, the results obtained could be explained by the traditional stress–strain curves of plasticized PVC films. The stress–strain curves of plasticized PVC films are similar to rubbers [41]. According to the stress–strain curves, higher values of elongation at break result in lower values of modulus at 100% if the tensile strength values are similar. Hence, the resultant values of modulus at 100% of samples with ITO content are much higher than that of control. Also, the higher values of elongation at break of samples with ATO content lead to the lowest values of modulus at 100%. With regard to the tearing property, the results are around 50 kN/m. Compared to control, a little enhancement can be observed. ITO or ATO particles absorb the plasticizer, which results in the flocculation phenomenon. Hence, the dispersion of ITO or ATO particles has been changed, and ITO or ATO particles assembled into larger size. Subsequently, the resultant cavities and air bubbles occur in the presence of local stress. Consequently, the presence of cavities and air bubbles results in the toughing effect, which explains the improvement of tearing property.

4. Conclusions

In this study, UV absorber, yellow pigment and ITO or ATO particles were all adapted to reach the target of harmful light blocking. UV absorber and yellow pigment can shield the light from 200 to 400 nm in UV region and 400 to 500 nm in high-energy visible wavelength, respectively. ITO or ATO particles give an excellent performance of NIR light (800–2600 nm) blocking. Significantly, ITO particles show stronger NIR light blocking performance than ATO particles, accompanied with higher visible light transmittance. The temperature test highlights great heat-insulation property of ITO and ATO particles to dissipate the heat accumulation. Moreover, the plasticized PVC matrix is reinforced by ITO or ATO particles to some extent. Results were further extended by completing similarly successful mechanical properties experiments. In conclusion, the polymer-inorganic hybrid thin films fabricated in this study possess high transmittance in visible wavelength combined with high shielding property in UV bands, blue light range and NIR spectral region.

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