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Influence of Flame Retardants TPP and PX200 in Combination with Zinc Borate in Bisphenol-A Polycarbonate/Acrylonitrile-Butadiene-Styrene Blends

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Abstract

Effects of the phosphates ester triphenyl phosphate (TPP) and phosphoric acid 1, 3-phenylenetetrakis (2, 6-dimethylphenyl) ester (PX200) with and without zinc borate on the flame retardancy mechanisms of bisphenol-A polycarbonate/ acrylonitrile-butadiene-styrene (PC/ABS) blends were investigated. The thermal decomposition was studied by thermo gravimetric analysis (TGA) and the flammability by LOI, UL 94 and smoke density rating (SDR). The thermal deformation was investigated using a heat distortion temperature instrument. The results show that PX200 make better performance than TPP in the decomposition process and forming char. Though TPP and PX200 in PC/ABS blends lead to the V-0 classification in UL94 and an obvious growth in limiting oxygen index (LOI), both of them have higher smoke density rating than that of neat PC/ABS. Combining Znb with TPP and PX200 respectively, T_{onset1} and T_{onset2} shift to high level, residue goes up and a clear synergy in LOI, whereas in UL94 classification they become less effective than PC/ABS +TPP or PC/ABS+PX200. 2wt% Znb can react to TPP or PX200 and promote blends to from char, thus decrease the SDR slightly. The heat deflection temperatures reveal that both TPP and PX200 have an inferior effect in PC/ABS blend, which due to the bad heat resistance of the flame retardant. The mechanical properties show that the dispersal of TPP and PX200 in PC/ABS is poor, which leads to a drop in elongation at break and notched charpy impact strength.

1. Introduction

Polycarbonate (PC)/acrylonitrile–butadiene–styrene (ABS) blends have been reported a lot because of their excellent mechanical and electrical properties and high heat distortion temperature. PC/ABS is a commercial polymer which is widely used as engineering thermoplastics for electronic engineering and other applications[1]. Due to the addition of ABS, the flame resistance of PC/ABS is worse than pure PC. In some applications such as electronic engineering, PC/ABS blends need the addition of flame-retardant (FR) compounds to decrease their flammability.

Halogen-containing compounds especially bromine and chlorine compounds are commercial flame retardants since their higher efficiency over other flame-retardants[2]. The

use of halogen-containing compounds has been restricted in consideration of the environment. During the decomposition of halogenated FR additives, toxic gases and high levels of smoke are released, which will cause secondary damage to the environment and human[3]. Currently, studies of halogen-free flame retardants are the favorable trends in either academia or industry[4]. Among these halogen-free flame retardants, much attention has been paid to phosphates ester such as triphenyl phosphate(TPP), bisphenol-A bis (diphenyl phosphate)(BDP), resorcinol bis (diphenyl phosphate)(RDP), hydroquinone bis (diphenyl) phosphate (HDP) and phosphoric acid 1,3-phenylenetetrakis(2,6-dimethylphenyl) ester (PX200).As we all know, BDP and RDP are liquid at room temperature, it's hard for us to blend with PC/ABS in particle. So in this work, we mainly compared the efficiency of powder TPP, PX-200 and their synergists for PC/ABS blend.

It is suggested that phosphate ester flame retardants mechanism is a complex process, and is generally considered to be both condensed-phase retarding mechanism as well as gas flame retardant mechanism of the collaborative process[5].Triphenyl phosphate (TPP) was the first generation of aryl phosphates used. It was used in cellulosic materials and polyurethane foams as a flame retardant[6, 7]. A hybrid system of nanoclay and triphenyl phosphate (TPP) was studied in E. Feyz's work. They found that the volatilized TPP played a major role in the gas-phase flame-retardation effect which was showed in cone calorimetrytesting[8]. Their conclusions were similar with Pawlowski et al. Pawlowski investigated the efficiency of TPP, RDP and BDP in PC/ABS blends [9, 10]. It was concluded that TPP acts mainly in the gas phase through flame inhibition, due to its high volatility and low decomposition temperature. RDP and BDP act mainly through flame inhibition, but also through some charring in the condensed phase. PX200 is a solid non-halogen phosphoric ester fire retardant, which has good machinability, unique advantages of self-extinguishing and good compatibility[11].
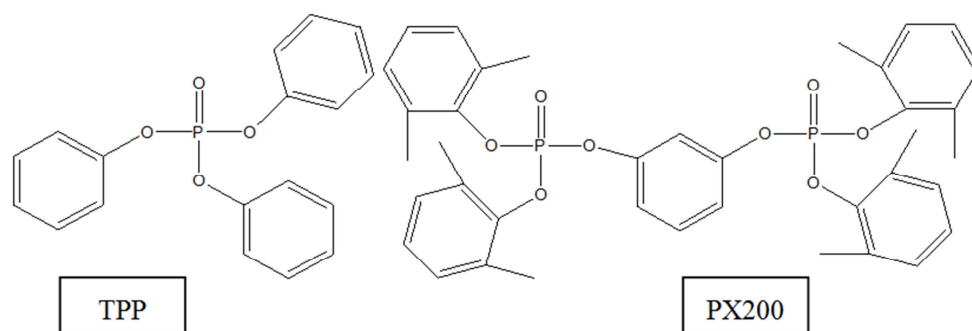
Recently, different flame retardants or flame retardants with adjuvant is combined to satisfy the increasing demands on efficiency of flame retardancy and combinations of properties. These retarded systems can obtain new, superior synergisms. Zinc borate (Znb) is often used as smoke suppressant to combine with phosphate ester. Because phosphates ester in PC/ABS blends usually increase smoke release[9]. Besides, Znb also can form an inorganic layer to prevent heat and combustible gas from releasing.

To obtain efficient flame retardant and smoke suppression of PC/ABS alloy, we studied the flame retardancy effect of TPP and PX200 and their retarded systems with Znb in PC/ABS blends. The focus was research on the performance of effects of TPP and PX200 in PC/ABS blends. Besides, the interaction of TPP or PX200 with Znb was also studied.

2. Experimental

2.1. Materials

PC (L1250Y) was from TEIJIN polycarbonate LTD (Janpan) and ABS (PA-758) was from CHIMEI (Taiwan) with melt flow index (MFI) (200 °C, 5kg) of 3.0 g $(10 \text{ min})^{-1}$. Styrene-maleic anhydride (SMA) was supplied by Sinopec Shanghai Petrochemical Company Limited (China).The halogen-free FR used in this research was additive-type triphenyl phosphate (TPP), and phosphoric acid 1,3-phenylenetetrakis(2,6-dimethylphenyl) ester(PX200), which were all from Daihachi Chemical Industry CO., Ltd. Phosphorus content of TPP was 9.5wt% , and PX200 was with 9wt% phosphorus content. The structure of TPP and PX200 is shown inFig.1.

Polycarbonate and Acrylonitrile-Butadiene-Styrene was delivered in the form of pallets while mica was in the form of powder.

Fig. 1. Structure of TPP and PX200.

2.2. Compounding Procedure

A commercially available weight ratio 80:20 of PC and ABS was used in the experiment.

PC and ABS were dried for 4 h respectively at 120℃ and 80℃. Then, the mixes were melt-blended according to the formulations given in Table 1 using a co-rotating twin-screw extruder (ZSK-25WLE) with L/D ratio of 40. The screw speed was 120 rpm and the temperatures of the extruder zones were adjusted from 200℃ to 240℃. After extruding, the resulting pellets were dried at 80℃ for 8h and then injection molded (JPH-10) into various shapes corresponded to the characterizations discussed below. The injection temperature profile was adjusted from 220℃ to 250℃.

In order to compare the efficiency of TPP and PX200 we use the same content of phosphorus in flame-retarded PC/ABS. For example, the content of phosphorus in 19wt% TPP and 20wt% in PX200 are equal. The composition of PC/ABS/TPP composite and PC/ABS/PX200 composite are given in Table 1.

2.3. Characterization

2.3.1. Thermal Stability and Flammability Testing

Thermo gravimetric analysis (TGA) was performed on TGA Q500 (TA) apparatus to investigate the thermal decomposition of the PC/ABS blend under N_2 atmosphere. Samples of 5 mg were heated from 50℃ up to 700 ℃ using heating rates of 10℃·min⁻¹, with a controlled dry nitrogen flow of 40 cm³·min⁻¹.

The UL-94 vertical burning test was carried out on 3.2 mm thick sheets according to GB/T 2408-1996.This test provides qualitative classification of the samples through measurement of the ease of burning or extinguishment of a polymer sample.

The Limiting oxygen index (LOI) of the formulations was determined with an LOI instrument (JF-3, made by Nanjing Jiangning Analytical Instrument Factory, China) on 80* 10* 4 mm³ sample sheets according to GB/T 2406—1993. The test is based on the lowest oxygen gas concentration that still sustains combustion of the sample. This test is not directly relevant to a natural fire situation but provides numerical values for research and quality control. In order to overcome problems of poor repeatability of data, ten or more specimens

for each sample were used.

The smoke density was measured according to GB/T8627-2007 by using a smoke density chamber (JCY-2, made by Nanjing Jiangning Analytical Instrument Factory, China). The size of specimen for the measurement was 25*25*4 mm³. The maximum smoke density was measured from the peak maxima of the curve of the light absorption time. Total amounts of smoke present in the chamber for 4 min time. We use smoke density rating (SDR) to judge the smoke density.

Heat Deflection Temperature (HDT) was measured by using a heat distortion temperature instrument (ZWK Test, made by MTS, China,) according to GB/T 1634-2004. The specimen size was 80*10*4 mm³. Samples were heated from 0℃ up to 200℃ using heating rates of 120℃·h⁻¹, with a controlled weight of 306kg. The displacement limit was 0.34mm.

2.3.2. Mechanical Testing

Tensile strength and percentage tensile elongation at break (ultimate elongation) were determined at 25 ℃ using a universal testing machine (LR30K, Ametek Co., England) according to Chinese standard GB/T1040-2008. And the tensile speed was 50 mm·min-1. Notched Charpy impact strength was carried out at 25 ℃ using a resil impactor (Ceast Co, Italy) according to Chinese standard GB/T1843-2008. Five specimens of each composition were tested and the average values were recorded.

Table 1. Compositions of the Formulations.

Material	PC (wt%)	ABS (wt%)	SMA (wt%)	PTFE (wt%)	TPP (wt%)	Znb (wt%)
PC/ABS	76.50	19.15	4	0.35	0	0
PC/ABS+19%TPP	62.12	15.53	4	0.35	19	0
PC/ABS+20%PX200	60.52	15.13	4	0.35	20	0
PC/ABS+17%TPP+2%Znb	62.12	15.53	4	0.35	16	2
PC/ABS+18%PX200+2%Znb	60.52	15.13	4	0.35	18	2

3. Results and Discussion
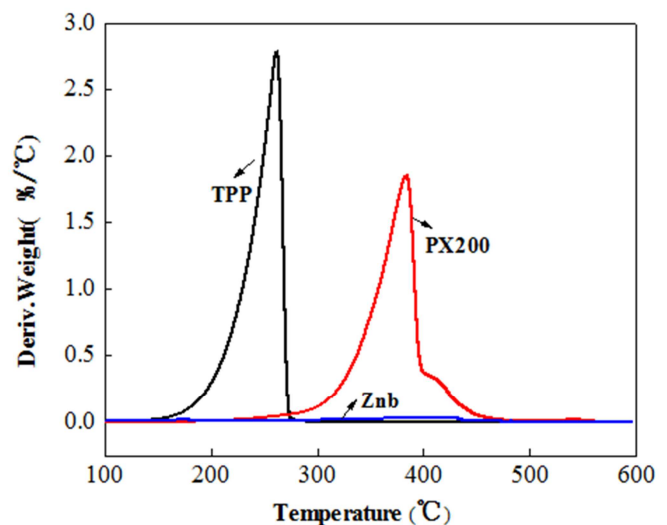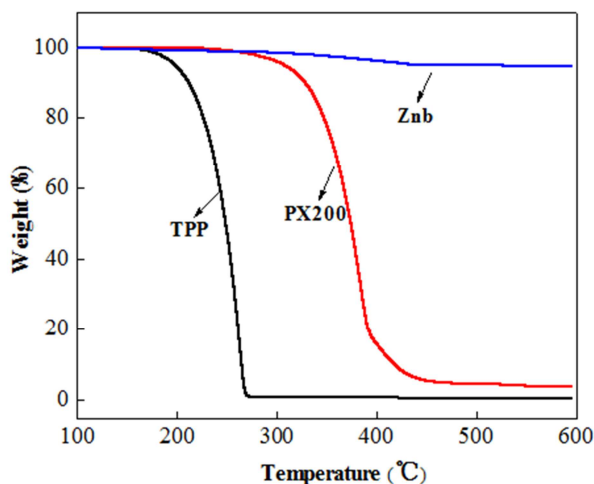
3.1. Thermal Decomposition

Fig. 2. TGA and DTG curves of the TPP、 PX200 and Znb in N_2 atmosphere.

TGA measurements on the blends were performed to

understand the process of thermal degradation in combustion. Thermal degradation of flame retardant TPP、PX200 and Znb are shown in Table 2 and Fig.2. The results of pure PC/ABS, PC/ABS+TPP, PC/ABS+TPP+Znb, PC/ABS+PX200 and PC/ABS+PX200+Znb are summarized in Table 2 and the corresponding masses are shown in Fig.3. All the measurements were in a heating rates of $10℃·min^{-1}$, with a controlled dry nitrogen flow of $40\ cm^3·min^{-1}$.

Table 2. Thermal analysis of flame retardant TPP、PX200 and Znb.

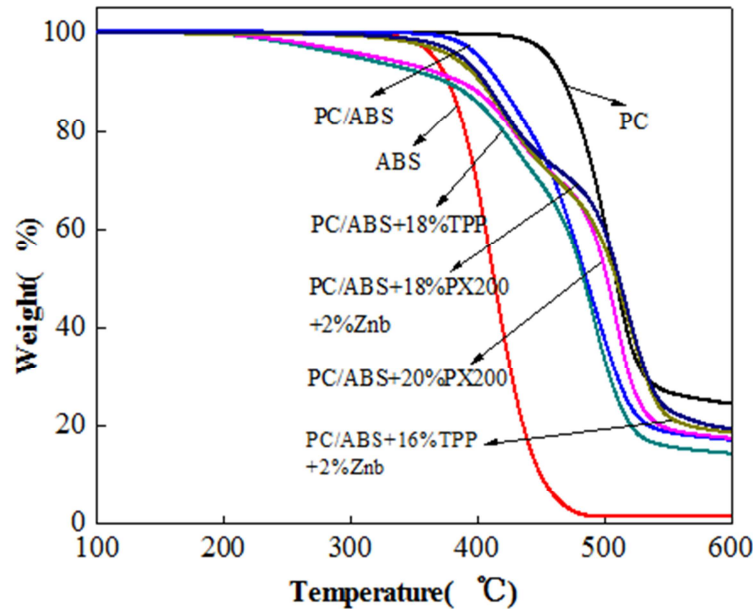| Flame Retardant	$T_{2wt\%}(℃)$	$T_{onset}℃$	$T_{50wt\%}(℃)$	Residue (wt%)
TPP	182.05	231.22	248.82	0.49
PX200	275.87	347.39	374.25	3.9
Znb	340.58	378.04	-	86.64

Fig. 3. TGA curves of the pure PC、ABS and flame-retarded PC/ABS in N_2 atmosphere.

Table 3. Thermal analysis of PC/ABS and flame retardant PC/ABS blends.

Material	$T_{2wt\%}(℃)$	Mass Loss I(ABS)		Mass Loss II(PC)		$T_{50wt\%}(℃)$	Residue at 600℃(wt%)
		$T_{onset1}(℃)$	$ML_1\ (\%)$	$T_{onset2}(℃)$	$ML_2\ (\%)$		
PC	448.16	-	-	478.31	-	508.81	24.39
ABS	357.56	384.36	-	-	-	413.38	1.39
PC/ABS	382.44	398.43	32.10	469.18	52.63	478.16	15.27
PC/ABS+19%TPP	233.04	365.81	35.10	484.10	49.61	488.90	15.29
PC/ABS+17%TPP+2%Znb	258.86	380.87	33.16	484.58	48.24	493.92	18.60
PC/ABS+20%PX200	340.31	374.72	30.48	489.60	52.66	498.63	16.96
PC/ABS+18%PX200+2%Znb	365.48	386.50	27.16	503.89	53.46	514.11	19.38

As shown in Table 2 and Fig.2, TPP、PX200 and Znb decompose in one step under nitrogen. TPP decomposes at a lower temperature thanPX200, with the T_{onset} 116.17℃ below that of PX200 (231.2℃ for TPP, 347.39℃ for PX200). TPPandPX200 leave only 0.5 to 4 wt% of residue, while Znb leaves more than 85 wt% of residue. That's because Znb is an inorganic mineral, it's more stable than organisms like TPP and PX200. For TPP and PX200, the core structure (Fig.1) has an influence on the thermal stability of the flame retardant[12]. As seen by the temperature at 2 wt% of weight loss ($T_{2wt\%}$) of 182.05℃for TPP, 275.87℃ for PX200. The decomposition of TPP starts 93.82℃ below that of PX200.During the decomposition, Znb starts to release water of crystallization in its structure above 300 ℃. As shown Table 2, Znb releases 13.36% water of crystal water. After that the decomposition of Znb stops.

The addition of ABS to PC changes the decomposition behavior of the components significantly. At the TGA curves of Fig. 3, one can see that pure PC/ABS and flame-retarded PC/ABS both decompose in two steps. While neat ABS and neat PC decomposes in a single decomposition step. According to neat PC/ABS, the first step mainly corresponds to the decomposition of ABS with a mass loss of 32.10%, which is higher than the content of ABS shown in Table 1. Onset temperature is 398.43℃, it is close to that of neat ABS. The second process relates to PC with the onset temperature 469.18℃, slightly lower than pure PC. PC is a naturally charringmaterial[13], and so 24.39 wt% residue is obtained at 600℃. ABS almost decomposes completely at 600℃. As to PC/ABS blend, the residue is higher than neat ABS and lower than neat PC. The entire phenomenon indicates that during the decomposition there is an interaction between PC and ABS,

which influences both the release of ABS and PC decomposition. This result is similar with the work of Perret B and his co-workers[14].

The decomposition temperature of the flame retardant is a main parameter enabling a condensed phase interaction with PC decomposition products. When TPP,PX200 are added to the PC/ABS blends, the thermal decomposition still occurs in a two-step process, but the decomposition temperatures are shifted compared to non-flame retarded PC/ABS blends (Table 3 and Fig.3).Overall, TPP has a worse impact on decomposition temperatures than PX200. At the beginning of decomposition, TPP apparently lower the temperature of 2wt% mass loss from 382.44℃ to 233.04℃, while PX200 shifts the temperature to 340.31℃. This is because the initial decomposition temperature of TPP and PX200 is 170 ℃ and 260℃ (Table 2and Fig.2), which is much lower than PC/ABS blends. The T_{onset1} decreases slightly to 365.81℃ in PC/ABS+19%TPP and 374.72 ℃ in PC/ABS+20%PX200 (398.43 ℃ for neat PC/ABS).However, the T_{onset2} are both increase near 20 ℃, 484.10 ℃ for PC/ABS+19%TPP and 489.6 ℃ for PC/ABS+20%PX200. At the first mass loss step, because of the entirely decomposition of TPP, the mass loss (ML_1) of sample PC/ABS+19%TPP is the most in the samples. Nevertheless, with 49.61% mass loss (ML_2), sample PC/ABS+19%TPP owns a lower mass loss than that of PC/ABS+20%PX200.The$ML1$ of PC/ABS+20%PX200are shifted downward, from 32.10% for neat PC/ABS to 30.48%, while the ML_2 increases to 52.66%. The amounts of residue for PC/ABS+19%TPP have no change, and for PC/ABS+20%PX200 are 16.96%, corresponding to an increase of 1.7%. Hence the flame retardant exhibits a condensed phase action in the blend, increasing the formation of char from PC/ABS. This interaction is lower for TPP due to its earlier decomposition/volatilization. As discussed above, the decomposition process of TPP is from 150 ℃ to 300 ℃, which of PX200 is from 200 ℃ to 500 ℃. Thus decomposition of TPP relates to the decomposition of ABS, PX200 relates to the decomposition of both PC and ABS. It

is assumed that aryl phosphate such asPX200 could decompose in the earlier stage to form phosphoric acid, and this acid could catalyze polymer matrix to dehydrate and etherify in order to form char. Further aryl phosphate takes part in the cross-linking reactions and become a part of the char [5,15]. Consequently, this char can act as a barrier, shielding the unburned polymer matrix against heat as well as hindering the diffusion of pyrolysis gases.

During the decomposition, Znb results in only a significant release of water (around 13.36 wt% in Table 2and Fig.2). When added 2% with TPP and PX200 in PC/ABS blends, Znb acts as an assistant flame-retardant. For PC/ABS+17%TPP+2%Znb the mass loss of two decomposition step are both decreased slightly (1.94 wt% for first and 1.37wt% for the second decomposition step) compared to PC/ABS+19%TPP without Znb (Table 3), considering that for PC/ABS+17%TPP+2%Znb less polymer decomposes(3.3 wt %) than in PC/ABS+19%TPP.The mass loss of the first decomposition step of PC/ABS+18%PX200+2%Znb is similar with PC/ABS+17%TPP+2%Znb, but the second become higher than PC/ABS+20%PX200. It may be because that PX200 reacts instead of PC with some of the water released by Znb. Hence the mass loss in the second decomposition is high. Totally, the amount residue of PC/ABS+18%PX200+2%Znbis up(by 2.42wt%). The additional of Znb make the $T_{2wt\%}$、T_{onset1}、T_{onset2} and$T_{50wt\%}$dramatically shift to high level. Comparing the data observed, a significant interaction between TPP, PX200 and Znb became obvious. Most probably alternative reactions occurred to the reaction of TPP, PX200 with PC and the water release of Znb and PC.

3.2. Variation of Fire Behavior

The flame retardancy of all samples with and without flame retardant was characterized by LOI, UL-94 methods and the smoke density chamber. The results for flammability are summarized in Table 4.

Table 4. The results for flammability.

Material	LOI(%)	UL94(3 mm)				SDR (%)
		Rating	$t_1(s)$	$t_2(s)$	dropping	
PC/ABS	22.4	NR	26.7	>50	No	90
PC/ABS+19%TPP	28.5	V-0	4.13	3.30	No	95
PC/ABS+20%PX200	29.3	V-0	2.97	1.32	No	92
PC/ABS+17%TPP+2%Znb	30.0	V-1	6.51	19.93	No	88
PC/ABS+18%PX200+2%Znb	32.5	V-1	6.27	15.34	No	85

Neat PC/ABS exhibits a LOI value of 22.4%, mainly because the addition of ABS. When TPP and PX200 are added to PC/ABS blend, the LOI value sharply goes up to 28.5% and 29.3%.The fire performances in the case of PC/ABS+19%TPP are similar in comparison with PC/ABS+20%PX200.PC/ABS achieved HB classification in the UL 94,with the burning time longer than 50s. Adding TPP and PX200 to PC/ABS yielded V-0 for 3mm thick samples, with almost immediate self-extinction after removing the

burner. Compare the after-flame time after the first ignition (t_1) and the after-flame time after the second ignition (t_2) of sample PC/ABS+19%TPP and PC/ABS+20%PX200, it can be seen that $t_2$is always shorter than t_1. It indicates that, TPP and PX200 are good for forming char after the ignition. Thus the char could protect the matrix from burning.

Keep the total amount of additives and substitute TPP or PX200 with Znb, LOI value of both PC/ABS+17%TPP+2% Znb and PC/ABS+18%PX200+2% Znb increase slightly. And

both show V-1 for the 3mmthick samples, because of burning time slightly longer than 10 s. Thus combining TPP or PX200 and Znb revealed a slight antagonism with respect to UL 94.

As we know neat ABS will generate a large amount of black smoke when burning[16]. So PC/ABS had a high smoke density rating (SDR) of 90%. The SDR increases with the addition of TPP and PX200. But the growth rate with PX200 is less than that of TPP. Probably because TPP hardly has a condensed phase action, which can from char to isolate combustible gas and smoke.

In comparison with PC/ABS with PX200 alone, we know that PC/ABS filled with 18%PX200 and 2%Znbhas significantly effects on the SDR properties. The SDR of PC/ABS+18%PX200+2%Znb can decrease to85%, which is 5% less than that of neat PC/ABS blend. Because Znb can work with PX200 promote blends to from char, thus isolate combustible gas to release. On the other hand, it can evaporate water to dilute the gases when polymer burns. In general, Znb can use as a smoke suppressant to combine with phosphate ester in PC/ABS.

3.3. Variation of Heat Deflection Temperature (HDT)

The HDT can be considered as a measure of the temperature at which certain creep compliance is reached after the polymer has been subjected to a standard temperature program[17]. The heat deflection temperatures (HDT) of blends are collected in Table 5.

With the addition of TPP and PX200, the HDT of blends sharply decrease from 103℃ to 72.0℃ and 78.8℃. The sudden drop may be due to the poor heat resistance of TPP and PX200.The effect of 2wt% Znb in combination with TPP and PX200 in PC/ABS is not very pronounced in enhance the heat deflection temperature. The heat deflection temperature of PC/ABS+TPP+Znb slightly increases to 78.1℃and PC/ABS+PX200+Znb to 85.9℃. As for HDT, in general, the reduction of PX200 in PC/ABS is less than that of TPP.

Table 5. *Heat Deflection Temperature of the blends.*

Material	HDT(℃)
PC/ABS	103
PC/ABS+19%TPP	72.0
PC/ABS+20%PX200	78.8
PC/ABS+17%TPP+2%Znb	78.1
PC/ABS+18%PX200+2%Znb	85.9

3.4. Variation of Mechanical Properties

The results of mechanical tests and comparison diagram are showed in Table 6.

It can be observed that the tensile strength progressively decreases as the addition of TPP and TPP +Znb, with value of 49.6MPa and 50.1MPa. An increase is observed with PX200in tensile strength, while it reduces a little to 54.0MPa when combining with Znb. The elongation at break of PC/ABS blends with TPP, PX200 and TPP+Znb, PX200+Znb decrease to around 60%.

The level of notched charpy impact strength for both flame-retarded PC/ABS blends is rather poor. Due to their incompatibility, the flame-retarded PC/ABS blends exhibit impact strength even lower to the ones showed by pure PC/ABS blend. The observed negative deviation is due to the poor interfacial adhesion between the PC/ABS and flame retardant phases, which causes poor stress transfer between the matrix and the dispersed phase.

Table 6. *Mechanical properties of the blends.*

Material	Tensile Strength(MPa)	Tensile Elongation at break(%)	Notched Charpy impact strength (KJ·m^{-2})
PC/ABS	56.5	107	55.8
PC/ABS+19%TPP	49.6	63	35.0
PC/ABS+20%PX200	60.5	66	23.3
PC/ABS+17%TPP+2%Znb	50.1	60	29.6
PC/ABS+18%PX200+2%Znb	54.0	62	20.5

4. Conclusion

The influences of TPP and PX200 with or without zinc borate are investigated in PC/ABS based on blends with the same phosphorus content. On the basis of the experimental works and test results, the following conclusions are drawn:

During the decomposition, TPP and PX200 with or without Znb shift the onset temperature (T_{onset1}) of first decomposition step to lower and the second decomposition step to higher temperature, and an increase in residue amount. Interactions in the condensed phase between aryl phosphates and decomposition products of PC are enhanced and lead to increased charring. The hybrid of TPP or PX200andZnb, especially for PX200 and Znb, also enable an enhancement for the thermal stability of the PC/ABS composites.

TPP and PX200 have presented an obviously growth in LOI values and V-0 classification, while Znb+TPP and Znb+PX200 exhibit an antagonist effect in the polymer matrix through UL-94 test by V-1 classification. Furthermore, because of the high content phosphorus TPP and PX200 both release a lot of smoke, the SDR is higher than that of neat PC/ABS. Combining TPP and PX200 with 2wt% Znb in PC/ABS leads to a reaction between both additives, and thus make the SDR lower than blends with TPP or PX200 alone and even lower than that of neat PC/ABS alloy.

For HDT, both TPP and PX200 with or without Znb reduce a lot to a lower level than neat PC/ABS. The sudden drop in elongation at break and notched charpy impact strength indicate that the dispersal of TPP and PX200 is bad.

Comparing all the results investigated above, synergy of flame retardancy between PX200 and Znb is a little more

efficiency in the PC/ABS blends than TPP with Znb. Combination Znb with PX-200, blends of PC/ABS could get V-1 classification in UL94 test, and a lower SDR and higher HDT than PC/ABS/PX200 blends. However, more work should do to improve the dispersal and thermal deformation of flame-retarded PC/ABS blends. Because the decrease in properties is an important disadvantage for the practical application of flame-retarded PC/ABS blends.

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