Properties of Poly(vinyl chloride) and Polyesteramides Blends

Radka Kalousková, Ludik Markvart, Jana Kredatusová, Jiří Brožek

Department of Polymers, Institute of Chemical Technology, Prague, Technická 5, 166 28 Prague 6, Czech Republic

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ABSTRACT: Blends of poly(vinyl chloride) (PVC) and polyesteramides (PEA), based on ε -caprolactam and ε -caprolactone, were prepared to improve the notch impact strength of PVC. Miscibility of components was assessed from glass transition temperature and is limited by the amount of PEA in the blend and by the content of ε -caprolactam structural units in PEA. The effects of the content and type of PEA on their thermal stability and notched impact strength were also investigated. The impact strength of the PVC blends reaches its maximum at an

equimolar lactam to lactone units ratio in PEA; it decreases with increasing content of PEA in the blend. An admixture of a small amount of poly(ε-caprolactam) as a compatibilizer made it possible to prepare PEA-rich PVC blends, exhibiting twice as high notch impact strength, as compared to pure PVC. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 826–833, 2009

Key words: PVC blends; polyesteramides; miscibility; thermal stability; toughness

INTRODUCTION

Poly(vinyl chloride) (PVC) exhibits many exceptional properties and finds utilization in many applications. During the processing, however, it is necessary to use a number of additives, able to improve the thermal and environmental resistance of the blend, to ease the processing, or to modify the utility properties of the material.^{1,2} In addition to strength, toughness belongs to the most important material properties as well. Due to the phase structure, the value of notch impact strength of PVC is not very high (especially at lower temperatures), which limits its use in some applications. The solution to the problem lies either in use of vinylchloride copolymers or in the preparation of blends of PVC and polymers having low glass transition temperature. Polymers used as modifiers, due to their limited miscibility with PVC, affect the microstructure of the blends, which leads to a substantial increase of their notch toughness.^{1–3} Most frequently, elastomeric materials (such as butadiene-acrylonitrile rubber, chlorinated polyethylene, ethylene-vinyl acetate copolymer, or acrylate elastomers), copolymers with a

two-phase structure (such as methyl methacrylatebutadiene-styrene or acrylonitrile-butadiene-styrene), are applied as modifiers.^{2–6} To increase the interphase compatibility, a suitable compatibilizer may be added to the system.

To modify the properties of PVC, polyamides or polyesteramides (based on ε -caprolactone and lactams) can also be used.^{7–10} Poly[(ω -laurolactam)*block*-(ε -caprolactone)] has successfully been applied as a compatibilizer of the PVC-polyamide 12 blends or the PVC/PP blends.^{8,9} The ester blocks of the copolymer lead to the PVC phase, as a result of a generally good miscibility of poly(ε -caprolactone) (PCLO) with this polymer, while the polyamide blocks are oriented to the polyamide phase. Thus a better connection of both phases is established on the interface, which improves the mechanical properties.

Biologically degradable statistical copolymer poly[(ε -caprolactam)-*co*-(ε -caprolactone)] [below denoted as polyesteramides (PEA)], prepared by an anionic polymerization of ε -caprolactone (CLO) and ε -caprolactam (CLA), is another interesting polyesteramide.^{11–13} If the sodium salt of CLA^{11,12} is used, PEA is formed with 60% yield and the unreacted low-molecular-weight fractions must be extracted. In contrast, ε -CLA magnesium bromide enables one to prepare PEA within the whole concentration range of the CLA structural units and with high a yield as 95%; moreover, any extraction of low-molecular-weight fractions can be omitted.¹³ In both cases, the copolymers have statistical microstructure: only one glass transition temperature was

Correspondence to: R. Kalousková (radka.kalouskova@ vscht.cz).

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always detected in the differential scanning calorimetry (DSC) curves, the value of which depended on the copolymer composition and was in good agreement with that calculated from the Fox equation.¹⁴ Dependence of melting enthalpy of PEA, and thus the content of crystalline phase on PEA composition, passes minimum at content of 30% lactam structural units in PEA.¹³

Blends of PVC and a statistical PEA-CLA/CLO copolymer, studied so far, were prepared by mixing tetrahydrofuran solutions of both polymers at 35° C.¹⁰ Glass transition temperature (T_{g}) determined by DSC served as a measure of miscibility of the components. Tg decreased with increasing PEA content, regardless of the CLA/CLO structural units ratio in PEA. Unlike the PVC/PEA blends modified by PEA with 36.6 mol % CLA, the blends having a lower content of the CLA units in PEA (i.e., 12.8 mol %) were homogeneous and the results of thermal measurements correlated well with the theoretical dependence calculated according to the empirical Fox equation. However, the solubility of PEA in tetrahydrofuran is limited by the content of the lactam structural units: only those PEA that contain less than ca. 20 mol % CLA units are soluble.¹⁰ Moreover, the preparation of PVC blends from solution is only scarcely used in practice: blending of polymers prevails.

The subject of the present article is the preparation of blends of PVC and a statistical polyesteramide consisting of ϵ -CLO and ϵ -CLA to improve the toughness of PVC.

EXPERIMENTAL

Materials

PVC Neralite 682 suspension type, Kh = 68, was kindly supplied by from Spolana Neratovice (Czech Republic). The heat stabilizer Tinstab MB360 (di-*n*-butyltin maleate) was purchased from Akcros Chemicals, GB, and dosed in an amount of 1.5 wt parts per 100 wt parts of PVC. Lubricant Bralen SA 200-22 (LDPE) (Slovnaft, Slovak Republic) was dosed in an amount of 1.0 wt part per 100 wt parts of PVC.

Poly[(ε -caprolactam)-*co*-(ε -caprolactones)] (PEAs) were prepared in Ref. 13 (i.e., by the anionic copolymerization of CLA and CLO) initiated by 1 mol % CLA magnesium bromide at 150°C (polymerization time 1 h), basic characterization of copolymers used is summarized in Table I.

Preparation of the blends

Blends of PVC and PEA in the form of foils 0.6- to 0.7-mm thick were prepared on a Polymix 80T two-roll mill, roller size 80/300 mm, slip 1.2 (rotation ve-

 TABLE I

 The Characteristic of Used Polyesteramides (PEA)¹³

[CLA] ₀ (mol %)	T _m (°C)	$\Delta H_{\rm m}$ (J g ⁻¹)	T _g (DMA) (°C)	T _g (DSC) (°C)	η_{red} (cm ³ g ⁻¹)
97	211	81	_	48	_
80	177	43	25	25	285
70	143	47	15	7	280
60	121	22	2	-4	254
50	93	16	-10	-19	263
40	76	27	-23	-33	203
30	51	11	-30	-40	201
20	55	43	-25	-51	172
10	59	62	-33	-52	134
0*	69	105	-44	-57	117

[CLA]₀ = initial concentration of ε -caprolactam in polymerization mixture; $T_{\rm m}$ = melting temperature; $\Delta H_{\rm m}$ = enthalpy of fusion; $T_{\rm g}$, glass transition temperature; $\eta_{\rm red}$ = reduced viscosity in tricresol (at concentrations 2.0–2.2 × 10^{-3} g cm⁻³, 25°C).

locity ratio 22 : 19). Rolling temperature was 180° C \pm 0.1°C; blend preparation time 7–8 min (optimum period of time with respect to the thermal stability and the notch impact strength of blends). The specimens were obtained by 5 min compression at 202°C and 30 MPa.

Procedures

PVC is inherently unstable; HCl is eliminated from polymer backbone and conjugate double bonds are formed during thermal treatment.¹⁵ That is why the processing of PVC requires the presence of the thermal stabilizer, which in addition to the inhibition of dehydrochloration, reacts with eliminated HCl and chlorides are formed. HCl that does not react with heat stabilizer any more and is released from the sample (referred to as "liberated HCl") was quantified by continuous potentiometry.¹⁶ The thermal stability (TS) values were read from the kinetic curves obtained for the individual blends. The TS value represents the time interval (in minutes) from the start of the heat treatment to the moment of a rapid increase of the amount of liberated HCl.

The color stability of the blends in the foils treated at 180°C in air by using TESTER TOM Model 204 was evaluated visually.

Two methods were used to determine T_g of blends. The DSC measurements were performed by using DSC-Q100 Modulated DSC (TA Instruments) in the temperature range 20–150°C with a constant heating rate of 10°C min⁻¹ and nitrogen purge (50 cm³ min⁻¹). The dynamic mechanical analysis (DMA) measurements were performed by using DMA DX04T tester (RIM, Czech Republic), in a temperature range between -20 and 150°C at a heating rate of 3°C min⁻¹; bending mode single cantilever with constant deformation of ± 0.2 mm and sinusoidal force at a frequency of 1 Hz was used.

Notched impact strength (a_k) was measured with the Charpy pendulum on specimens (60 mm × 6 mm × 4 mm) with an average notch depth of 1 mm at 23°C.

The morphology of some PVC/PEA blends was characterized by using a scanning electron microscope (SEM). The blended samples were frozen in liquid nitrogen and then fractured. The fracture surfaces were gold-plated by sputtering and surface changes were observed using TESLA BS 340 at accelerated voltage 15 kV.

RESULTS AND DISCUSSION

Statistical PEA, the melting temperatures of which did not exceed the processing temperature, having 80 mol % of lactam units at the most (Table I), were used to prepare the blends. PEA applied (containing 20–80 mol % of lactam structural units) excels in high notch impact strength: They are unbreakable at room temperature.¹³ It could thus be expected that their presence in PVC would increase its toughness.

Effect of structure of PEA on properties of the PVC/PEA blends

PVC-PEA miscibility

First, the effect of the PEA structure on the properties of blends prepared was studied. In addition to a stabilizer and a grease, the blends contained 5 wt parts PEA per 100 wt parts of PVC, the content of the CLA structural units in PEA ranging from 0 to 80 mol %. PEA having a low content of the lactam units (up to 30 mol %) and a low melting temperature was easily blended with PVC on cylinders. Miscibility of the blends worsened with increasing content of the CLA structural units in PEA, which manifested itself by enhanced stickiness/adhesion of the blends to the cylinders. Blends of PVC and PEA with 70 to 80 mol % CLA could no more be removed from the cylinders in the form of compact foils (note that polyamide 6 is immiscible with PVC).

Only one T_g was identified on the DSC curves for all of these blends (Fig. 1). In comparison with pure PVC ($T_g = 87.8^{\circ}$ C), values of T_g for the blends of PVC and PCLO, as well as of PVC and PEA with 10–20 mol % of the CLA structural units, are distinctly lower. This indicates a single-phase system, in which PEA acts, to a certain extent, as a plasticizer. With increasing content of the lactam, the plasticizing effect of PEA decreases, but T_g of the blend increases more steeply than would correspond to the theoretical dependence predicted by the Fox equation.¹⁴ Above 30 mol % of the CLA structural units



Figure 1 Dependence of glass transition temperature (T_g) obtained from DSC (Δ) or calculated according to the Fox relation (\diamond) and dependence of notched impact strength (a_k , \bullet) of PVC/PEA blends (with PEA content 5 wt part per 100 wt part) on CLA structural units in PEA.

in PEA, T_g of the blend is close to that of PVC alone. This suggests that, at a higher content of the lactam units in PEA, both components are blended imperfectly. T_g of the polyesteramide component, due to its low content (4.6 wt %), was detected neither by DSC, modulated DSC (not shown here), nor DMA.

Mechanical properties

Notch impact strength of the PVC/PEA blends (a_k) containing 5 wt parts of PEA changed with increasing content of the lactam units in the structure of PEA (Fig. 1) and reached its maximum value (5.6 kJ m⁻²) for the equimolar composition of PEA (1 : 1 CLA:CLO mole ratio). As compared to PVC alone ($a_k = 3.7$ kJ m⁻²), there was an increase by ca. 50%. Apparently, in this blend, an optimum dispersion of PEA in PVC was achieved, which was influenced by relatively low melting temperature and low degree of crystallinity (Table I). The drop of a_k for samples having a higher content of CLA was probably caused by deteriorating the homogeneity of the blends, in which PEA forms domains without any reinforcing effects.

Thermal stability

The processing of PVC requires the presence of the thermal stabilizer, which inhibits dehydrochlorination of the blend during the thermal strain. If certain organotin compounds are used for this purpose, labile C—Cl bonds on the polymer chain are stabilized by formation of a complex with the not fully coordinated tin atom and the release of HCl from PVC is



Figure 2 Dependence of the amount of liberated HCl in PVC and PVC/PEA blends on degradation time at 180°C in air. The blends contain 5 wt parts PEA per 100 wt parts of PVC, differ in CLA/CLO structural units in PEA (in the legend CLA/CLO in mol % and thermal stability in minutes).

hampered. In addition to that, the stabilizer binds released HCl, the autocatalytic activity of which is thus limited. Moreover, the stabilizer, as a derivative of maleic acid, can react (through the Diels-Alder reaction) with conjugated double bonds, formed by the dehydrochlorination of the polymer; thus, such an interruption of the long polyene sequences may contribute to a long-term preservation of the original color of the blend.¹⁷ On the other hand, however, it is known that the presence of PCLO in PVC affects the efficiency of the organotin stabilizer. Polyesters interact noncovalently with α -hydrogen on the PVC chain and thus block the formation of the stabilizing complex with the Sn atom of the stabilizer.¹⁸

Although the stabilizer binds released HCl, its effect on releasing HCl from the PVC/PCLO blends is smaller than for PVC alone (Fig. 2). The result is that the number of short polyene sequences on the PVC chain sharply increases and thus the color of the blends deepens (Figs. 3 and 4). Besides, the stabilizer, as a derivative of maleic acid, may be deactivated by a reaction with end groups of PCLO (and predominantly of PEA), which manifests itself by a deterioration of TS (Fig. 2) and a darkening of the blends. The determination of concentrations and types of the end groups in PCLO and PEA is difficult and no attention is paid to it in the literature. With regard to studies on blends of PVC and aliphatic-aromatic copolyesters based on recycled polyethyleneterephtalate and CLO¹⁹, it can be assumed (by analogy) that, using another stabilizing system (based on, e.g., Ca/Zn or Pb), the negative effect of PEA on the thermal and color stability of the PVC blends could be reduced.

Undoubtedly, also the modifier itself (that is, PEA), containing polyamide sequences sensitive to increased temperature and presence of air (Fig. 4), contributes to the yellowing of the blend.

The dependence of the thermal stability of the PVC/PEA blends on the composition of PEA passes through a minimum (Fig. 2). The increase of TS of the blends containing PEA with more than 50 mol % CLA may be related to the limited miscibility of the components. These PEAs, apparently due to the phase separation in the mixtures with PVC (cf. values of T_g of the blends which are identical with those of PVC), do not react with the stabilizer to such an extent that would influence the rate of dehydrochlorination.

The effect of the content of PEA on the properties of PVC/PEA blends

The next part of the study is devoted to the effect of the content of PEA on the properties of PVC/PEA blends. The blends contained 0, 2.5, 5, 10, 15, 16.4, and 20 weight parts of PEA per 100 weight parts of PVC. PEAs containing 10, 50, and 80 mol % CLA units were used to study the concentration dependencies, and PCLO was used for the sake of comparison.

It is apparent from Figures 4 and 5 that the thermal stability of all blends distinctly decreases with increasing content of PEA. This is in agreement with the above conclusion because the increase of the concentration of PEA causes an increase of the component, which reduces the efficiency of the organotin thermal stabilizer.



Figure 3 Dependence of the absorbance increase of the band at 267 nm (ΔA) of PVC and PVC/PCLO blend (5 wt parts PCLO per 100 wt parts PVC) on the degradation time (at 180°C in air).



Figure 4 Color stability of samples from PVC, PVC/PEA, and PVC/PCLO blends subjected to heat treatment at 180°C in air (shifting rate, 2 mm min⁻¹). Number of mixture-concentration PEA in wt parts/100 wt parts PVC; content of CLA/CLO structural units in PEA in mol %: 1–5, 0/100, 2–5; 10/90, 3–5; 20/80, 4–5; 30/70, 5–5; 50/50, 6–0, 7–2, 5; 10/90, 8–5; 10/90, 9–10; 10/90, 10–16,4; 10/90.

Changes of T_g in dependence on the PEA concentration in the blends are closely related to the content of the lactam structural units in PEA. If PEA with a plasticizing effect (i.e., that containing 10 mol % CLA units or PCLO was used), only a single T_{gy} the value of which decreased with increasing content of the additive in the blend, was detected

(Fig. 6). The T_g values of the blends (determined by both DSC and DMA) are identical to those calculated from the Fox equation, which confirms the miscibility of the components.

Plasticizing PVC by polyesteramide (PEA, with CLA/CLO 10/90) slightly restrains a_k (Fig. 7),



Figure 5 Dependence of thermal stability (TS) at 180° C in air of PVC /PEA blends on content of PEA in blends (PEA with various content of CLA/CLO structural units: 0/100, 10/90, 50/50, and 80/20).



Figure 6 Dependence of glass transition temperature (T_g) of PVC blends (solid line, DSC or DMA; dashed line, calculated from the Fox relation) on amount of PEA with 10 mol % CLA structural units (determined by DSC) or PCLO (determined by DMA) in blends.



Figure 7 Dependence of notched impact strength (ak) of PVC/PEA mixtures on PEA content in blend and different content of CLA/CLO structural units in PEA (10/90, 50/ 50, and 80/20).

despite of the fact that the component added exhibits a high value of a_k^{13} and is fully miscible with PVC. We assume that this phenomenon relates to the already known migration of the plasticizer on the basis of CLO to the surface of the PVC/PCLO blend.¹⁸ The subsequent crystallization of the released PCLO undoubtedly influences the impact strength of the blend. This process is accelerated by the presence of organotin stabilizer, as mentioned above.

By contrast, two values of T_g were revealed in the DMA record for the PVC blends containing more than 10 weight parts of PEA with 50 and 80 mol % CLA structural units in the chain; these values correspond to individual components (Table II). This indicates an immiscibility of PVC and PEA on the molecular level. DMA records of some blends are given in Figure 8.

The immiscibility of the components affects a_k of the blends in dependence on the content of PEA in the PVC/PEA blend (Fig. 7). While a_k of PVC plasticized by PEA with 10 mol % CLA decreases almost linearly with increasing concentration of the addi-

TABLE II Glass Transition Temperature of PVC/PEA Blends (with 80 and 50 mol % CLA Structural Units) Determined by DMA

	*T _g (°C)			
wt Parts of PEA per 100 wt Parts of PVC	PEA (80 mol % CLA)	PEA (50 mol % CLA)		
0	97 (-)	97 (-)		
2.5	97 (-)	Not prepared		
5	96 (-)	96 (–)		
10	95 (-)	94 (-29)		
15	89 (22)	95 (-29)		
20	89 (19)	Not prepared		



Figure 8 Dynamic-mechanical analysis of PVC and PVC/ PEA blends. Content of CLA/CLO structural units in PEA and amount of PEA in the blends (in wt parts per 100 wt parts of PVC) are in the legend.

tive, notch impact strength of the PVC/PEA blends containing 50 or 80 mol % CLA units in the copolymer chain passes through a maximum at 5 weight parts of PEA/100 weight parts of PVC. As was already mentioned above, higher values of notch impact strength of PVC/PEA blends having the equimolar CLA/CLO ratio are related to their better dispersibility. Undoubtedly, the drop of a_k with increasing PEA content is connected with increasing heterogeneity of both blends; for a sufficient amount of the additive, this heterogeneity manifests itself through the existence of two T_g values (Table II and Fig. 8). As compared to other blends, the PVC/PEA (CLA/CLO 50/50) blend is rather exceptional due to



Figure 9 Dependence of thermal stability (TS), glass transition temperature (T_g , DSC), and notched impact strength (a_k) of PVC/PEA/PCLO blends on PCLO content in the blend, content of PEA(with 80 mol % CLA structural units) is 5 wt part per 100 wt part PVC.

Figure 10 Dependence of notched impact strength (a_k) of PVC/PEA/PCLO blends on content of PEA (with 50 mol % CLA structural units) in the blend. Content of PCLO in blends is 0.2 wt part per 100 wt part PVC.

the fact that, with increasing PEA content, the transparency of the blend is improved and the surfaces of the rolled foils become very markedly smoother.

The effect of PCLO on the miscibility of PVC and PEA

In an attempt to improve the miscibility of the phases and, consequently, to increase the toughness of the PVC/PEA blends, we admixed a small amount of PCLO, which is used as a polymeric plasticizer of PVC and belongs to polyesters well miscible with chlorinated polymers.¹⁸

It follows from Figure 9 that the presence of 0.2–2 weight parts of PCLO in the PVC blend containing 5 weight parts of PEA (CLA/CLO 80/20) does not



Figure 11 DMA of PVC and of PVC/PEA/PCLO blends, content of PEA (with 50 mol % CLA structural units) 10 or 20 wt parts per 100 wt parts of PVC and PCLO content 0.2 wt parts per 100 wt parts of PVC.

influence markedly the thermal stability and $T_{\rm g}$ and $a_{\rm k}$ remain almost unchanged up to 0.5 weight parts of PCLO; for a higher content of PCLO, $a_{\rm k}$ decreases by ca. 1 kJ m⁻².

Further, PVC blends containing 10 and 20 weight parts of PEA (CLA/CLO 50/50) and 0.2 weight parts of PCLO/100 weight parts of PVC were assessed. The addition of PCLO had a positive effect on a_k of the PVC/PEA blends (Fig. 10). At 20 weight parts of PEA per 100 weight parts of PVC, $a_k = 9$ kJ m⁻²



Figure 12 SEM micrographs of fracture surfaces of PVC/ PEA blends. Composition of blends: (A) 10 wt parts PEA (50 mol % CLA) per 100 wt parts of PVC; (B) 15 wt parts PEA (80 mol % CLA) per 100 wt parts of PVC; (C) 10 wt parts PEA (50 mol % CLA) + 0.2 wt parts PCLO per 100 wt parts of PVC.

was achieved, which is 2.5 times higher than that of pure PVC. Again, two values of T_g (indicating thus a heterogeneous blend) were detected in the DMA records; however, the broadening of the peak of the loss factor (tan δ), corresponding to T_g of PVC, suggests an improved mutual dispersion of PEA and PVC (cf. Figs. 8 and 11). We suppose that presence of PCLO in the blend would improve dispersibility of components on the interface and thus increase the a_k values.

By contrast, only a single T_g value was found even in this case by DSC (a finding similar to the case of uncompatibilized mixture of PVC with 15 weight parts of PEA), identical with T_g of PVC. Clearly, this method is insufficient for the characterization of blends of PVC and semicrystalline polymeric additives.

SEM images of PVC/PEA blends prepared in the presence or absence of PCLO unambiguously demonstrate their different structures (Fig. 12). Heterogeneity of the PVC/PEA (50 mol % CLA) blend is clear in micrograph (A) and as evidenced by the decreased a_k . The addition of a little amount of PCLO in this mixture (B) leads to finer and more regular dispersion of phases, which means to form a microheterogenous structure necessary to ensure good mechanical properties of the blend.

CONCLUSIONS

Miscibility of PVC and PEA was studied by assessing the DSC and DMA records. PEAs containing 10 or 20 mol % CLA structural units are miscible with PVC. Glass transition temperatures determined are in a good agreement with $T_{\rm g}$ s calculated from the Fox equation. With increasing content of PEA in these blends, the notch impact strength decreases.

PVC and PEA containing 30–80 mol % of the CLA units are immiscible; the notch impact strength passes through a maximum at the concentration of 5 weight parts of PEA in the blend; at this concentration, the highest values were achieved for an equi-

molar CLA/CLO structural units ratio in PEA. In this case, the dispersion of PEA in the blend attains its optimum. For further increasing the toughness of the PVC/PEA blends, it is possible to apply a higher content of PEA combined with a small amount of PCLO, which improves the miscibility of both components leading to a microheterogeneous two-phase

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