

Potentiometric studies on the influence of poly(N-vinylpyrrolidone) on the thermal degradation behavior of poly(vinyl chloride) blends

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Article Information

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The thermal degradation behavior of poly(vinyl chloride) (PVC) and poly(N-vinylpyrrolidone) (PVP) blends was investigated using potentiometric measurements of the released HCl gas during the degradation process, estimating the degree of discoloration of the degraded samples and measuring the thermal stability (T_s) values. The influence of the PVP percentage in the blend and, moreover, of the addition of commercial dibasic lead carbonate stabilizer to the blend was studied with regard to thermal stability. It turned out that the dehydrochlorination rate of the blend was promoted by increasing the PVP concentration in the blend.

PVC is a thermally unstable polymer if permanently exposed to temperatures higher than 60 °C. It undergoes rapid degradation already at higher temperature during the molding process, thus the life time of articles made from PVC is reduced under such conditions apart from plasticization effects. The thermal degradation consists of an autocatalytic dehydrochlorination reaction resulting in the release of hydrochloric acid and formation of conjugated double bonds [1]. The dehydrochlorination reaction and the associated formation of conjugated polyene sequences in the polymer molecule generate an unacceptable discoloration of the polymer [2].

Additionally, physical and mechanical properties of the polymer affect its applicability together with crosslinking and chain-scission at elevated temperatures, thus influencing the molecular weight of the polymer [2-4].

The presence of defects or thermally sensitive sites in the polymer chains is one of the main reasons why PVC has such a low

thermal stability. Generally speaking, a problematic site is the existence of tertiary carbon atoms (-CHCl-). Moreover, defect structures in PVC chains exist and are generated during synthesis and manufacturing, such as allyl double bonds with respect to chlorine (-CH = CH-CHCl-), double bonds as (terminal) end groups (-CH = CH₂), oxygen containing groups, in particular peroxy groups (C-O-O•/respective C-O-OH) and peroxide residues (C-O-O-C), head-to-head structures (-CHCl-CHCl-) and the steric orientation of the polymer (tacticity) [5-12].

Polymer degradation is initiated through such labile structures in the polymer chain associated with the propagation of hydrochloric release and the buildup of polyene sequences.

The thermal degradation behavior of PVC has been studied extensively by, e. g., Sabaa et al. [13-16] either in presence of organic or non-organic additives using the potentiometric measurement of the evolved HCl gas and by evaluating the discoloration

of the degraded samples. McNeil and co-workers [17-19] investigated the thermal stability of PVC blend systems by analyzing the volatilization products as well. Pearce et al. also reported on the thermal oxidation and photo-oxidation of some PVC blend systems [20]. Ultimately, Dong et al. have studied the thermal degradation of PVC-PVP blends using FTIR and Raman spectroscopy [21].

The literature reports further significant results on other properties of PVC blends [22, 23], for example, on the mechanical and thermo-oxidative behavior of PVC blends modified with epoxidized natural rubber (ENR) and acrylonitrile butadiene rubber (ABR) [24]. Mohamed et al. studied the thermal degradation behavior of PVC-poly(vinyl butyral) (PVB) blends using thermogravimetry. The thermal stability of such blends was improved by increasing the PVB percentage [14, 25]. The thermal stability of PVC-ethylene-vinyl acetate copolymer (EVA) blends was also investi-

gated [26]. All these blends showed autocatalytic effects in HCl release, thus the thermal stability was decreased. Generally, blends with EVA having higher vinyl acetate (VA) contents showed a reduced degradability in comparison to those with lower VA-containing EVAs. The thermal degradation of PVC-poly(methyl methacrylate) (PMMA) was slightly decelerated [27] and PVC-polyaniline (PANI) blends were also studied, with reactions of released hydrochloric acid with PANI observed (protonation) [28].

Another variant for improving the thermal durability of PVC is the addition of low-molecular weight organic and inorganic stabilizers. Thus, the thermal stability of PVC in presence of organic additives was investigated [29]. In particular, heavy metal-based stabilizers were considered.

However, layered double hydroxides (LDH) such as natural hydrotalcite or synthetic products containing Al and Mg have been proved to be favorable alternatives for heavy metal-based stabilizers due to their remarkably high capacity to react with HCl [30]. LDHs are non-toxic, environmentally friendly layered inorganic materials which are efficiently incorporated into the PVC resin to design translucent articles [31]. The effects of new synthetic LDHs, such as MgCuAl-LDH, MgFeAl-LDH and MgSbAl-LDH, were also studied [30, 32].

In this study, potentiometric measurements were used to investigate the thermal behavior of PVC and its blends with PVP in presence of dibasic lead carbonate (DBLC) as often tested standard stabilizer. Here, attention was directed to low PVP additions in the range from 0 to 5 % PVP based on the PVC weight to maintain the properties of PVC unaltered.

Experimental procedure

Materials. A PVC suspension was purchased from Hüls Company (Germany) with a K value of 70; dibasic lead carbonate (DBLC) from National Lead Company (Germany) were applied for this study. PVP was obtained from Sigma-Aldrich. Its average molecular weight (MW) amounted to $MW \approx 40\,000$ g/mol. Thermogravimetric analyses (TGA) of pure PVP samples were performed using the Shimadzu TGA 50H equipment at a heating rate of $10\text{ }^\circ\text{C}/\text{min}$ and a flow rate of $20\text{ ml} \times \text{min}^{-1}$.

Synthesis of PVC-PVP blend samples. PVC rigid samples were obtained in accordance with the following method:

PVC	1 g
DBLC	2 wt.-%
PVP	0 – 5 wt.-%

These three materials were carefully mixed in a mortar and 0.2 g of the resulting fine powder was applied for measuring the dehydrochlorination rate of PVC, PVC-PVP blends with and without DBLC. The results obtained were the average of three comparable experiments for each test.

Methods of evaluation of thermal degradation behavior. Evaluation of the efficiency of DBLC and PVP addition to PVC was performed by measuring the dehydrochlorination rate utilizing continued potentiometric measurements of the evolved hydrogen chloride gas. A comprehensive illustration of this method was presented elsewhere [33, 34].

A potentiometer (pH meter) consisting of a silver electrode and a saturated calomel reference electrode was applied. It was switched to a silver electrode and a saturated calomel reference electrode. The system

used for the potentiometric measurements was represented as scheme in Figure 1.

IR spectra were recorded in the range of wave numbers from 4000 to 400 cm^{-1} at $25\text{ }^\circ\text{C}$ using a Tescan Shimadzu infrared spectrophotometer (FTIR 8000, Japan).

Results and discussion

The thermogram of PVP demonstrates that the main decomposition step occurred at the 400 to $460\text{ }^\circ\text{C}$ temperature range with a weight loss of $\approx 80\%$ in air (see Figure 2).

The weight loss of the polymer blend in the range from room temperature to $250\text{ }^\circ\text{C}$ was attributed to the vaporization of low molecular weight oligomers [35] (see Figure 2). It must be considered that the high-molecular weight PVP was thermally stable up to $400\text{ }^\circ\text{C}$. Consequently, thermal decomposition of PVP should not principally superpose the PVC weight loss.

Despite of the higher thermal stability of PVP as given by the TGA curve, the results revealed a destabilization effect of PVP when blended with PVC either in presence or absence of DBLC as commercial stabilizer as described in the discussion below.

The rate of isothermal dehydrochlorination of rigid PVC at $180\text{ }^\circ\text{C}$ in air with different concentrations of PVP and each 2 wt.-% of dibasic lead carbonate (DBLC) as commercial thermal stabilizer is presented in Figure 3.

The results of a non-stabilized PVC blank sample and another PVC sample stabilized with 2 wt.-% of DBLC are presented for comparison. The results reflect a notable progress in the stabilizing efficiency of PVC by addition of DBLC in presence of PVP if compared to the blank PVC sample. However, the strongly increased stabiliz-

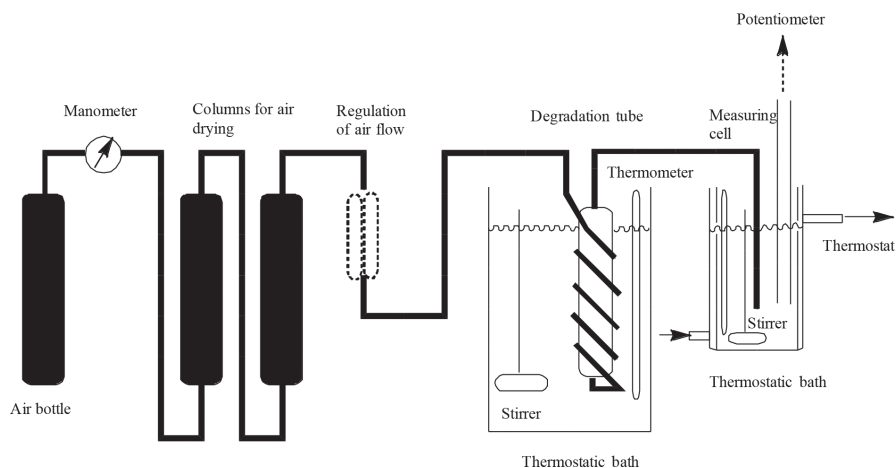


Figure 1: Schematic illustration of the apparatus used in the quantitative determination of HCl

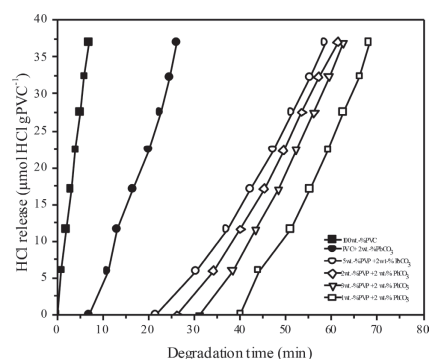


Figure 2: Thermogravimetric analysis (TGA) of PVP in air atmosphere

ing efficiency decreased with increasing percentage of PVP in the PVC blend.

The decrease of the stabilizing efficiency with increasing the percentage of PVP in the blend was illustrated by a reduction in the thermal stability values (Ts), i.e. the period during which no noticeable amounts of hydrogen chloride gas was released, and by an increase in dehydrochlorination rates during the degradation reaction (see Figure 3).

Figure 4 presents the dehydrochlorination rates of PVC samples blended with PVP (without DBLC) in various concentrations.

Samples of the non-stabilized PVC and PVC stabilized with 2 wt.-% DBLC were given for comparison. The results show a slight improvement in the Ts values if compared to that of pure PVC. Furthermore, the results also demonstrated the higher rate of dehydrochlorination of PVC if blended with 2 wt.-% PVP in comparison to the same concentration of DBLC (2 wt.-%). Dong et al. [21] have reported before that increasing admixing of PVP to PVC decreased the dehydrochlorination temperature. The rates of dehydrochlorination thus increased as a function of growing PVP concentration. However, the samples showed comparable Ts values if compared to that of virgin PVC as represented in Figure 4.

To suggest a possible mechanism for the thermal behavior of PVC-PVP blends, a series of additional experiments have been made and the structural changes in materials were characterized by IR spectroscopy of such blend samples that had been isothermally degraded in air at 180 °C, for the time interval from 10 to 60 min (see Figure 5).

An elevated concentration (5 wt.-%) was used in all experiments to favor the clear identification of any changes in the spectra. The degraded samples were extensively washed with water and alcohol to remove loosely bonded polar degradation products. The IR spectra were recorded from the degraded and the residual material of the filtrate after washing.

The results of isothermally degraded PVC-PVP (5 wt.-%) show the appearance of an absorption band in the region between 1580 and 1650 cm^{-1} in the IR spectrum, which corresponds to the degraded PVC associated with olefinic $>C=C<$ [36], and another band also at 1660 cm^{-1} which may be due to CO-NH of PVP (see Figure 5). The broadness of the band indicate the strong self-association of the amide group. The IR spectrum of the residue after washing with water is given in Figure 6.

It displays the presence of a new doublet at 3100 to 3200 cm^{-1} which may be attributed to the formation of $>N^+H Cl^-$ [37-39].

The effect of blending PVP in various concentrations to degraded PVC samples was visually noticeable by the level of discoloration corresponding to the degradation level in presence or absence of DBLC as is represented in Tables 1 and 2, respectively.

The results depict the high extent of discoloration of PVC-PVP samples either in presence or absence of DBLC if compared to the blank PVC. On the other side, PVC admixed with 2 wt.-% DBLC revealed a lower degree of discoloration as compared to PVC in absence of DBLC (see Table 2).

Additive concentration	Discoloration and degradation time (min)				
	0	10	20	30	45
Blank PVC	Colorless	Brown	Dark brown	Dark brown	Dark brown
2 wt.-% DBLC	Colorless	Buff	Buff	Orange	Orange
2 wt.-% DBLC + 1 wt.-% PVP	Colorless	Buff	Buff	Orange	Brown
2 wt.-% DBLC + 2 wt.-% PVP	Colorless	Buff	Buff	Orange	Brown
2 wt.-% DBLC + 3 wt.-% PVP	Colorless	Buff	Buff	Orange	Brown
2 wt.-% DBLC + 5 wt.-% PVP	Colorless	Buff	Buff	Orange	Brown

Table 1: Extent of discoloration of thermally degraded rigid PVC at 180 °C and in air as well as in presence of 2 wt.-% DBLC blended with various concentrations of PVP as a function of degradation time

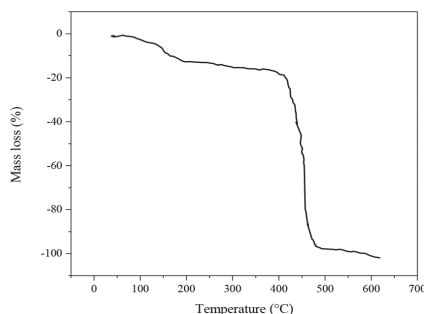


Figure 3: Rate of dehydrochlorination of rigid PVC enriched with 2 wt.-% DBLC commercial stabilizer blended with various concentrations of PVP at 180 °C in air

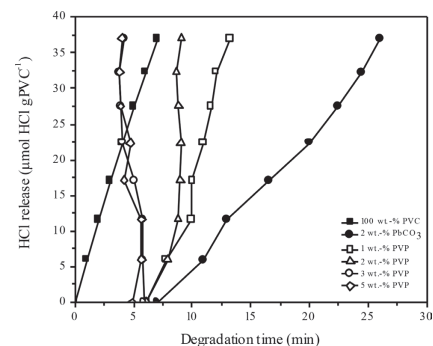


Figure 4: Rate of dehydrochlorination of rigid PVC at 180 °C and in air as well as in the presence of various concentrations of PVP

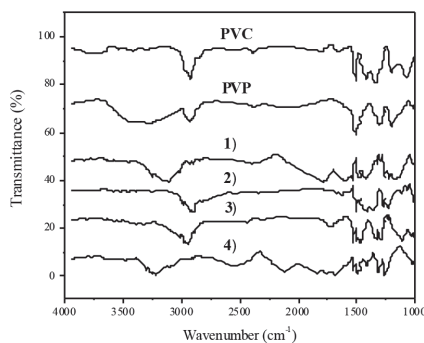


Figure 5: Changes in the IR spectra of PVC-PVP (5 wt.-%) samples degraded isothermally at 180 °C and in air as well as a function of degradation time, 1) 20 min, 2) 40 min, 3) 60 min, 4) 80 min

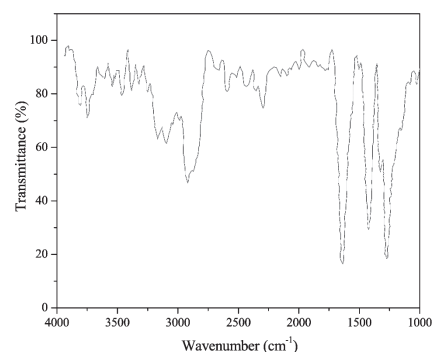


Figure 6: IR spectrum of the residue of degraded PVC-PVP (180 °C, in air)

Additive concentration	Discoloration and degradation time (min)				
	0	10	20	30	45
Blank PVC	Colorless	Brown	Dark brown	Dark brown	Dark brown
2 wt.-% DBLC	Colorless	Buff	Buff	Orange	Orange
1 wt.-% PVP	Colorless	Pale brown	Brown	Dark Brown	Dark brown
2 wt.-% PVP	Colorless	Pale brown	Brown	Dark brown	Dark brown
3 wt.-% PVP	Colorless	Brown	Brown	Dark brown	Dark brown
5 wt.-% PVP	Colorless	Brown	Brown	Dark brown	Dark brown

Table 2: Extent of discoloration of thermally degraded rigid PVC at 180 °C and in air as well as in presence of various concentrations of PVP as a function of degradation time

at 3000 to 3500 cm^{-1} after exposure to HCl, in comparison to that of untreated PVP.

This band may be overlapped with νOH and corresponds to the formation of $>\text{NH}^+\text{Cl}^-$ (see Figure 8). Furthermore, the elemental analysis demonstrated the presence of chlorine in the HCl treated PVP sample.

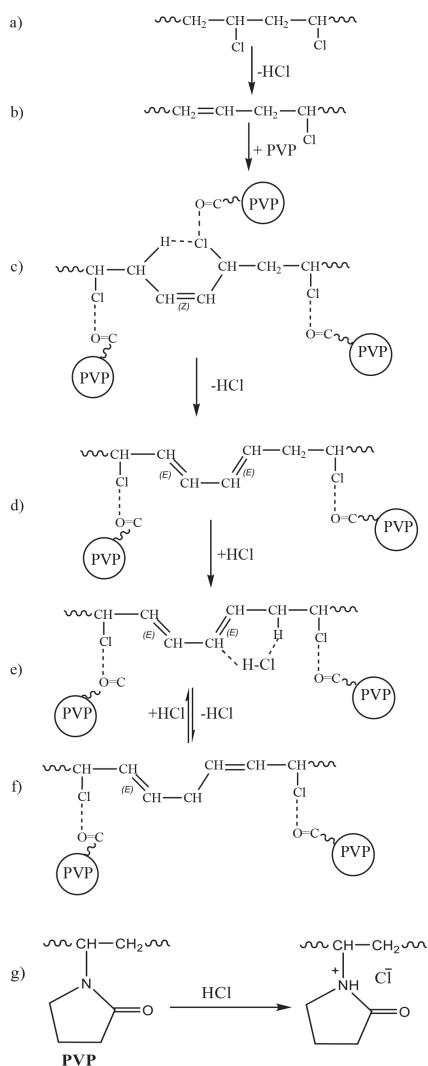


Figure 8: Degradation mechanism of PVC-PVP blends

From the above results, a probable mechanism of the interaction of PVC and PVP is presented below, adapting some details from Dong et al. [21] (see Figure 8).

A synergistic effect of the PVP and DBLC additives as commercial stabilizers was observed. The effect of the mixing ratio of PVP with DBLC was investigated in the range of 0 to 100 % PVP relative to DBLC. The overall mixed blend concentration was maintained constant at 2 wt.-% depending on the PVC weight. The results of the dehydrochlorination rate and the Ts values are depicted in Figures 9 and 10, respectively.

The results demonstrate the existence of a true synergistic influence resulting from the combination of PVP and DBLC, which reached its maximum when the two additives were mixed in 25 % PVP: 75 % DBLC ratio. A further evidence for the observed synergistic effect was obtained from the discoloration if the two additives (taken in various weight ratios) were mixed. A notable reduction in discoloration of the polymer was found for 0/100 or 25/75 % PVP/DBLC when heated up to 180 °C in air for 30 min (see Table 3).

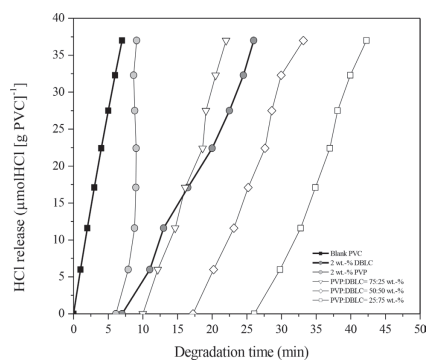


Figure 9: Rate of dehydrochlorination of rigid PVC at 180 °C and in air as well as in the presence of various weight ratios of PVP mixed with DBLC, the overall mixed stabilizers concentration was kept constant at 2 wt.-% based on the weight of PVC

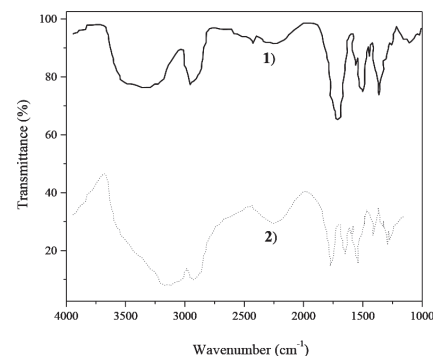


Figure 7: IR spectra of PVP, 1) before treatment, 2) after treatment with HCl gas at 180 °C for 30 min

Conclusions

Potentiometric measurements have proved that 5 wt.-% PVP addition increased the rate of dehydrochlorination. The reduction in thermal stability of the admixed PVP was illustrated by the increase in the degradation values (dehydrochlorination rate and Ts values) and also by the higher extent of discoloration as compared to the blank PVC or the reference commercial stabilizer when applied alone. However, blending PVP together with DBLC (25 : 75)

PVP/DBLC weight ratio	Discoloration
0/0	Dark brown
0/100	Orange
25/75	Orange
50/50	Brown
75/25	Dark brown
100/0	Dark brown

Table 3: Extent of discoloration of thermally degraded rigid PVC at 180 °C and in air as well as for 30 min in presence of various weight ratios of admixed PVP and DBLC

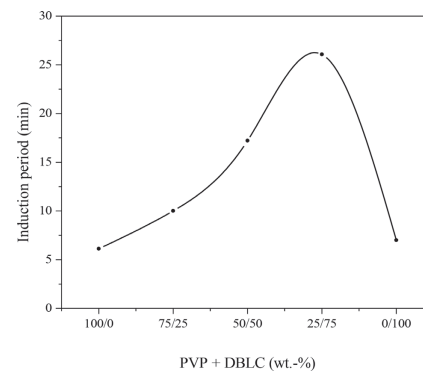


Figure 10: Thermal stability (TS) of PVC as indicated by duration of induction period in dependence on mixing ratio of PVP-DBLC additions

to PVC decreased the dehydrochlorination rate and the discoloration of PVC blends if compared to blank PVC. In an extended subsequent study, a layered double hydroxide (LDH), which acts as a secondary thermal stabilizer (HCl absorber) and a primary thermal stabilizer will be tested.

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