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THERMAL PROPERTIES OF POLYPROPYLENE POST-CONSUMER WASTE (PP PCW)

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Abstract

Differential scanning calorimetry has been used to study the heat flow during melting and crystallisation of a range of polypropylene post-consumer waste (PP PCW) grades and blends. The heat flow curves and the heat capacity curves indicated that the PP PCW grades and blends contained contaminants even after manual sorting and a cleaning process. The enthalpies of the PP PCW grades were lower than that for the virgin grades, as a result of degradation. Small amounts of polymeric contaminants (up to 10%) did not affect the enthalpies of PP PCW although other contaminants may have had some effect. The enthalpies of the PCW blends could in general be predicted by a linear additive rule, which is of importance for recycling a variety of PP PCW products.

Keywords: contamination, enthalpy, heat capacity, MDSC, polypropylene, recycling

Introduction

Polypropylene (PP) is an important commodity plastic and is used extensively for packaging consumer goods. With increasing use, the amount of discarded packaging material in the waste stream is also increasing. Management of polypropylene post-consumer waste (PP PCW) is therefore crucial and, recycling is a viable option for waste management. Recyclability of PP PCW is dependant on its structure and properties. The thermal properties, in particular, are of importance for semicrystalline polymers such as PP for an understanding of the crystallinity and the crystalline structure of the waste product. The structure and properties of PP PCW, however, have not received attention so far apart from studies of reprocessed PP. Thermal analysis, so far, have been restricted to reprocessed, artificially degraded [1, 2] and industrial waste PP (PP IW) [3].

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The crystallinity of PP depends on the molecular mass, the molecular mass distribution, the presence/absence of comonomers and additives/foreign particles, and on the processing history [4–9]. For reprocessed PP and PP exposed to ultraviolet radiation under laboratory conditions, crystallisation was reported to be influenced by the extent of degradation; both the degree of crystallinity as well as the crystalline form of PP were affected. For example, Rabello and White [2] observed that the melting enthalpy of PP increased on increasing the time of ultraviolet exposure up to a critical time but remained unchanged beyond this critical exposure time. This was suggested to be due to a competition between two aspects of degradation, namely, reduction in molecular mass and formation of chemical impurities such as carbonyl and hydroperoxide groups. At the initial stages the reduction in molecular mass was said to be more dominant and enthalpy increased but with longer exposure, crystallisation process was hampered by the presence of chemical impurities and therefore the enthalpy remained unchanged beyond the critical time. These researchers also observed that highly degraded PP crystallised as a mixture of α-PP and β-PP. A similar effect was also observed for mechanically stressed PP by Valenza and La Mantia [1]. Under normal circumstances, PP crystallises as monoclinic α phase; formation of hexagonal β phase occurs only under specific processing conditions and/or in the presence of specific additives and some pigments [7, 10, 11]. Since PP used in the studies by Rabello and White [2] and Valenza and La Mantia [1] did not contain any specific additives and were not prepared under any specific conditions, conducive of formation of β-PP, it is unclear as to the mechanism of formation of β-PP due to degradation.

PP PCW differs from reprocessed and artificially degraded PP in that PCW is exposed to a range of phenomena including thermal and mechanical loading during the manufacture, filling, transport, storage, use and disposal at the municipal recycling facilities (MRF). In addition, these materials also suffer from contamination [12, 13]. These factors are expected to affect the thermal properties of PP PCW.

The purpose of the present study was to investigate the thermal properties of PP PCW from kerbside collection of household waste. A range of PP packaging waste was collected from MRFs in Melbourne, Australia. The waste had therefore been exposed to realistic degrees of degradation and a variety of contaminants. These were manually sorted into three streams, processed and, subsequently, thermal properties determined to evaluate the effect of degradation and contamination on the properties. The effect of blending two or more of these PCW materials on the crystallisation characteristics was also investigated.

Experimental

Materials and processing

Three types of PP PCW packagings, ice cream containers (I), margarine containers (M) and fruit juice bottles (B), were obtained by manual sorting from two local MRFs in Melbourne, Australia. The typical contaminants present in the PP PCW included dirt, food remnants, glass pieces, fragments of bags and films and ice cream container lids. In the case of I and M, greasy food remnants were present and these resulted in sticking of bag and film fragments to the containers. The collected PP PCW was washed in hot caustic solution and granulated.

Flakes of PCW materials were extruded to form pellets for ease of subsequent injection moulding and, in the case of blends, for better mixing. This was carried out in an intermeshing counter-rotating Brabender DSK 42/7 twin-screw extruder, operating at 200–210°C. Injection moulding was done using an Engel CC90 injection moulding machine operating at 200–230°C, screw speed of 200 rpm, injection speed of 30 mm s^{-1} and post injection pressure of 50 bars. The mould temperature was maintained at 60°C.

The grades and blends studied and their melt flow indices are given in Table 1. The binary blends studied were I+M, I+B and M+B, containing equal parts by mass of the components, Table 1. A ternary blend W0 was prepared by mixing 33% each of I, M and B. A blend containing a known amount of polymer contaminant was also prepared (W10) which consisted of 30% each of I, M, B and 10% low-density polyethylene (LDPE) ice cream container lids, Table 1. Thermal analysis was also carried out for virgin PP grades equivalent to the PP PCW grades. The virgin grades studied are listed in Table 2.

Table 1 Materials – PCW grades and blends with their MFIs

	Material	MFI (g/10 min)
Grades	I (ice cream containers)	39.0
	M (margarine containers)	8.0
	B (fruit juice bottles)	2.0
Blends	I+M	17.0
	I+B	10.0
	M+B	4.0
	W0	6.5
	W10	7.2

Table 2 Materials – virgin grades with their MFIs

	Material	MFI (g/10 min)
Virgin	V (ice cream container grade)	23
	A (APD155 – margarine container grade)	2
	G (GE6100 – fruit juice bottles)	1

Differential scanning calorimetry (DSC)

A TA Instrument TMDSC model 2920 instrument was used for DSC. An intercooler cell was operated using nitrogen as the heat transformer gas with a flow rate of 100 mL min^{-1}. Helium with a flow rate of 25 mL min^{-1} was used as a purge gas. The samples were heated and cooled in the range 20–200°C using underlying heating and cooling rates of 2°C min^{-1}, a modulation period of 40 s and modulation amplitude of 0.3°C. Modulation mode of DSC was adopted in the present study since it yields accurate measurement of heat capacity. Polymer samples weighing 5–10 mg were cut from the moulded bars and used for DSC. Each sample was heated and cooled for two cycles. The first cycle was used to obtain thermal/processing history of the samples while the second cycle was used to obtain information related to the intrinsic polymer structure.

Heat flow signals recorded by the instrument were Fourier transformed to obtain the total heat flow and the heat capacities of the samples. The melting enthalpies were calculated by integrating the area under the peak of the endotherms (heating cycle) in the total heat flow curves. The upper limit of integration used was the end of the melting endotherms. The lower limit of integration used was the temperature at which the heat capacities measured on heating deviates from that measured during cooling. The crystallisation enthalpies were calculated by integrating the area under the peak of the exotherms (cooling cycle) in the total heat flow curves. For crystallisation enthalpies the lower limit of integration adopted was the same as that for the melting enthalpies while the upper limit of integration was that of the beginning of crystallisation [14]. The reproducibility of heat flow was 5%, consistent with the value reported by Cser *et al*. [15].

Results

Thermal properties of individual PP PCW grades

The typical total heat flow curves for I, M and B are shown in Fig. 1. In these figures, the solid lines represent the first cycle while the broken lines represent the second cycle. The melting peaks for all the PCW grades are around 160°C, and the crystallisation peak around 130°C, typical for α-PP, the most common crystal form of PP. A comparison of the cooling curves of the three grades show that the peak height of M is lower compared with I and B and the curve is wider than those for I and B. This indicates that the crystallisation of M was slower than the other two and occurred over a wider temperature range.

The total heat flow curves for I, M and B (Fig. 1) do not show any significant shift between 1st and 2nd cooling cycles. The peak crystallisation temperatures are also unchanged between the two cycles. These observations indicate that the PCW grades have consistent thermal stability.

The heat capacity curves during the heating cycle show some smaller peaks beside the main PP peak, Fig. 2; such peaks were also detectable in the total heat flow curves (Fig. 1). For I, M and B, the peaks are located around 120°C, 122°C and 128°C, respectively, Fig. 2. These peaks correspond to different forms of PE such as LLDPE and HDPE [14]. This is surprising because PE was not added intentionally to these grades. The presence of these small amounts of PE in PP PCW therefore indicates contamination in these materials. From the heat capacity curves of the three grades (Figs 2a–c) it appears that the heat capacity of M is higher than the other two grades. This is unexpected since this grade differs from the other two only by the

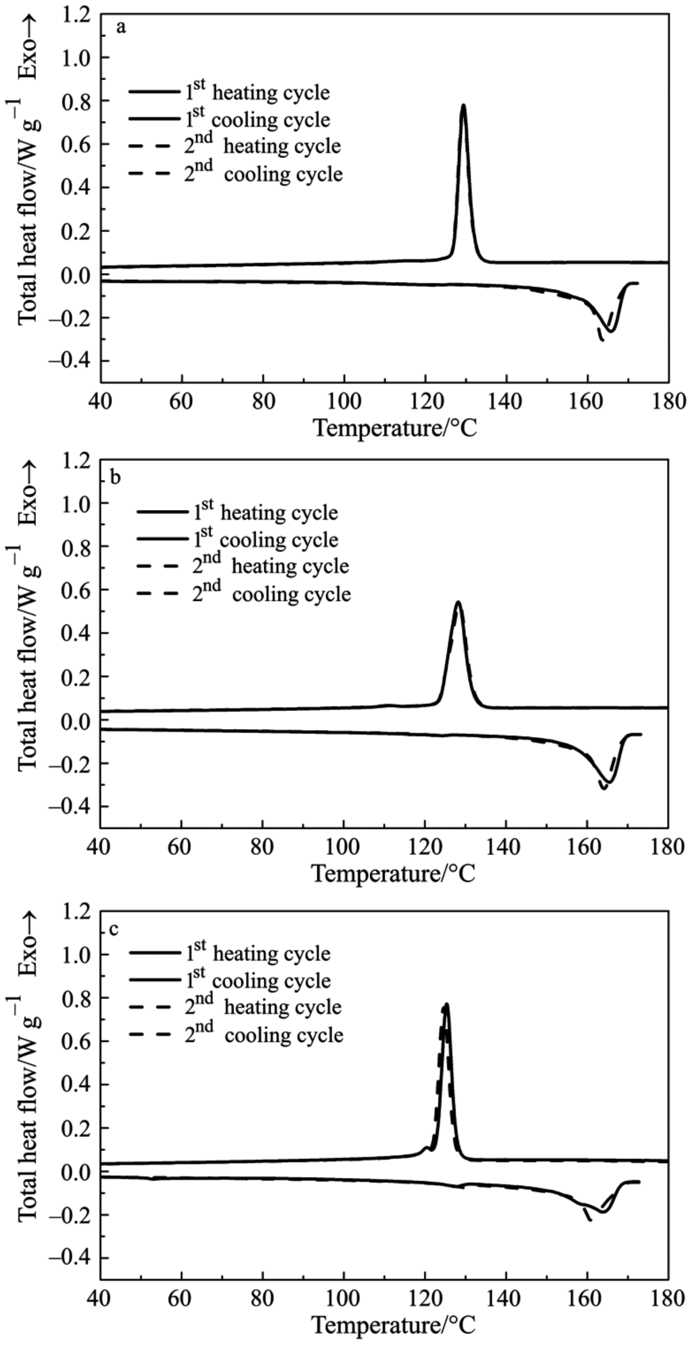

Fig. 1 Total heat flow curves for individual grades of PP PCW: a – I, b – M and c – B

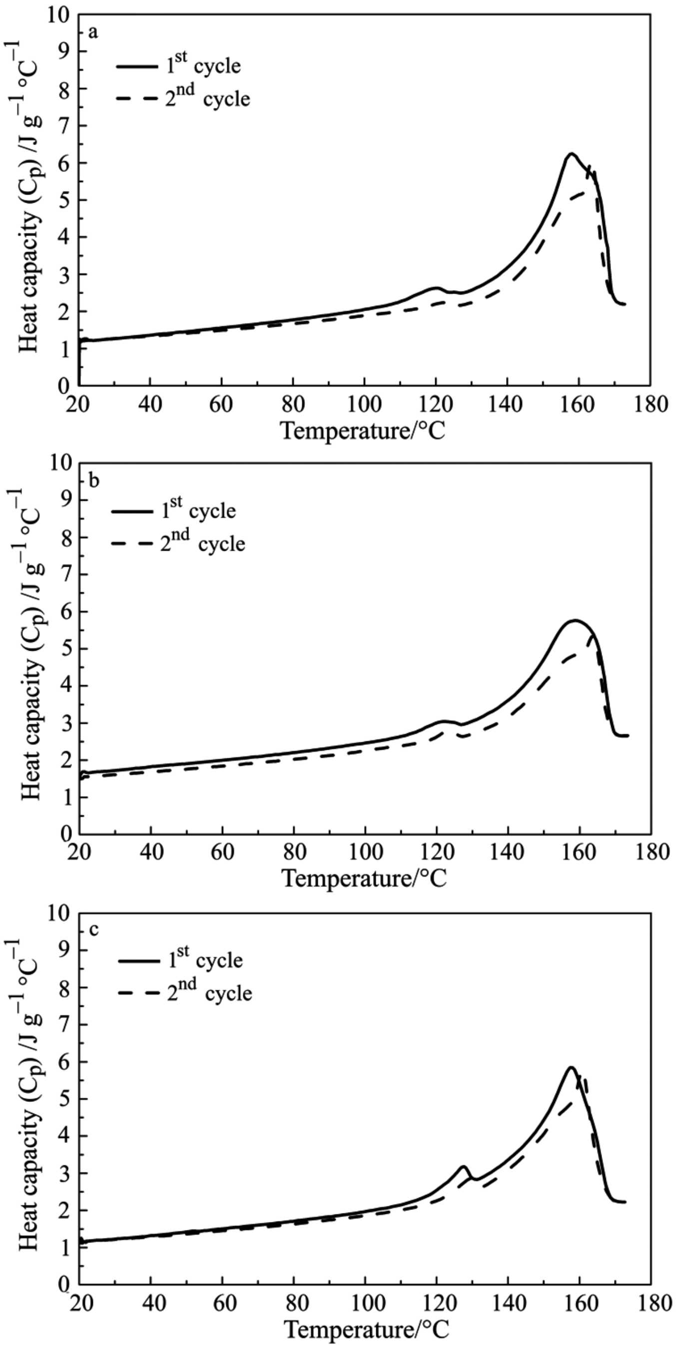

Fig. 2 Heat capacity curves for individual grades of PP PCW: a – I, b – M and c – B

presence of minor amounts of inorganic and organic pigments and acrylate based ink (about 2–3%). Such minor amounts of additives are not likely to affect the heat capacity of PP. The reason for the observed higher heat capacity of M is not apparent.

The enthalpy values for the individual grades of PP PCW during melting and crystallisation, calculated from the heat flow curves, are listed in Table 3. The intrinsic melting enthalpies ($\Delta H_{2\mathrm{melt}}$) for I and M are similar (74.5 ± 0.5 J g^{-1}) and higher than that for B (64.3 J g^{-1}). The intrinsic crystallisation enthalpies ($\Delta H_{2\mathrm{Cr}}$) for I and M were also similar (74.7 ± 0.6 J g^{-1}) and higher than that for B (68 J g^{-1}), Table 3. The MFI of the grade B was lower than those for I and M (Table 1) and therefore the enthalpies of B were also lower than I and M. The enthalpies of virgin grades V, A and G, equivalent to the PCW grades I, M and B respectively are listed in Table 4.

A comparison of the intrinsic melting and crystallisation enthalpies of PCW grades (Table 3) with those of the virgin grades (Table 4) indicated that in general, the melting and crystallisation enthalpies of the PCW grades were lower than those of the comparable virgin grades. The difference in enthalpies between the PCW and comparable virgin grades was greater for the high molecular mass (low MFI) grades than for the low molecular mass grades (high MFI), Tables 3 and 4. These differences are considered later (in ‘Discussion’).

Table 3 Changes in enthalpy of injection moulded individual grades of PP PCW

Grade	$\Delta H_{1\mathrm{melt}}$/J g^{-1}	$\Delta H_{2\mathrm{melt}}$/J g^{-1}	$\Delta H_{1\mathrm{cr}}$/J g^{-1}	$\Delta H_{2\mathrm{cr}}$/J g^{-1}
I	69.6	74.9	75.3	74.1
M	70.3	74.2	75.0	75.3
B	64.7	64.3	68.6	68.1

Table 4 Changes in enthalpy of injection moulded virgin PP grades

Grade	$\Delta H_{1\mathrm{melt}}$/J g^{-1}	$\Delta H_{2\mathrm{melt}}$/J g^{-1}	$\Delta H_{1\mathrm{cr}}$/J g^{-1}	$\Delta H_{2\mathrm{cr}}$/J g^{-1}
V	79.2	77.1	77.7	77.6
A	94.9	90.6	87.7	87.1
G	113.7	100.1	101.8	102.8

Thermal properties of PP PCW blends

The total heat flow curves for the PCW binary (I+M, I+B and M+B) and ternary (W0) blends are illustrated in Figs 3 and 4a. The melting and crystallisation peaks for the blends were similar to those for the individual PCW grades. As for the individual PCW grades, the cooling curves for the blends did not show any significant difference in peak temperatures between the two cycles. This indicates that similar morphologies were formed in both cycles.

The heat capacity curves of the PCW binary blends and the ternary blend (W0) are illustrated in Figs 5 and 6a. Both the total heat flow curves and the heat capacity curves of these blends show some smaller peaks in addition to the main PP peak. The peak temperatures of these smaller peaks are similar to those observed for the individual grades and correspond to LLDPE and HDPE. It is, therefore, apparent that the PCW blends show characteristics similar to the individual PCW grades.

In order to investigate the effect of PE contamination, a blend (W10) was prepared containing equal parts of the three PCW grades (I, M and B) with 10% PCW LDPE ice cream container lids (a typical contaminant, difficult to avoid during sorting at the MRFs). The total heat flow curve of W10 is shown in Fig. 4b. A comparison of the total heat flow curve of the ternary W0, and that for the blend containing additional 10% LDPE, W10, (Figs 4a and b) do not show any significant differences. A comparison of the heat capacity curves of W0 and W10 (Figs 6a and b) shows that the minor peak for W10 is broader than W0. The temperature range of this broad peak is similar to the temperature range for W0 with an additional peak at 110°C, corresponding to LDPE. This suggests that, in the present selection of PP PCW streams, contamination by LDPE has not occurred, i.e. LDPE lids were successfully removed by manual sorting.

The melting and crystallisation enthalpies of the binary blends, calculated from heat flow curves, are tabulated in Table 5. The intrinsic melting enthalpies ($\Delta H_{2\mathrm{melt}}$) of the binary blends are 80.3, 71.7 and 68.3 J g^{-1} for I+M, I+B and M+B, respectively, and 74.6 J g^{-1} for W0. The intrinsic crystallisation enthalpies ($\Delta H_{2\mathrm{Cr}}$) of the blends followed the same pattern as the melting enthalpies, Table 5. The melting and crystallisation enthalpies of the blend W10 are 77.6 and 72.9 J g^{-1}, respectively, Table 5. This is not significantly different than those for the blend W0.

The intrinsic enthalpies of the PCW blends were also calculated using the data for the individual grades and a linear additivity rule:

$$\Delta H_{\mathrm{blend}} = \sum_{\mathrm{i}} w_{\mathrm{i}} \Delta H_{\mathrm{i}}$$

where w_{i} is the mass fraction of the constituents of the blend and ΔH_{i} is the corresponding change in enthalpy.

The predicted enthalpies of the PCW blends are listed in Table 5. Generally, the measured values are similar to the predicted values with the exception of the blend I+M. The similarity in predicted and measured enthalpies of the blends suggests that the different PCW grades are miscible in each other and the crystallinity is additive. The reason for the difference between measured and predicted enthalpy for the blend I+M, however, is currently not apparent.

Table 5 Changes in enthalpy of injection moulded blends of PP PCW

Blend	$\Delta H_{1\mathrm{melt}}$/J g^{-1}	$\Delta H_{2\mathrm{melt}}$/J g^{-1}	Predicted $\Delta H_{2\mathrm{melt}}$/J g^{-1}	$\Delta H_{1\mathrm{cr}}$/J g^{-1}	$\Delta H_{2\mathrm{cr}}$/J g^{-1}	Predicted $\Delta H_{2\mathrm{cr}}$/J g^{-1}
I+M	76.2	80.3	74.6	78.5	80.9	74.7
I+B	70.0	71.7	69.6	73.3	72.3	71.1
M+B	68.1	68.3	69.3	69.1	70.7	71.7
W0	67.0	74.6	71.1	71.6	70.1	72.5
W10	70.9	77.6	–	73.4	72.9	–

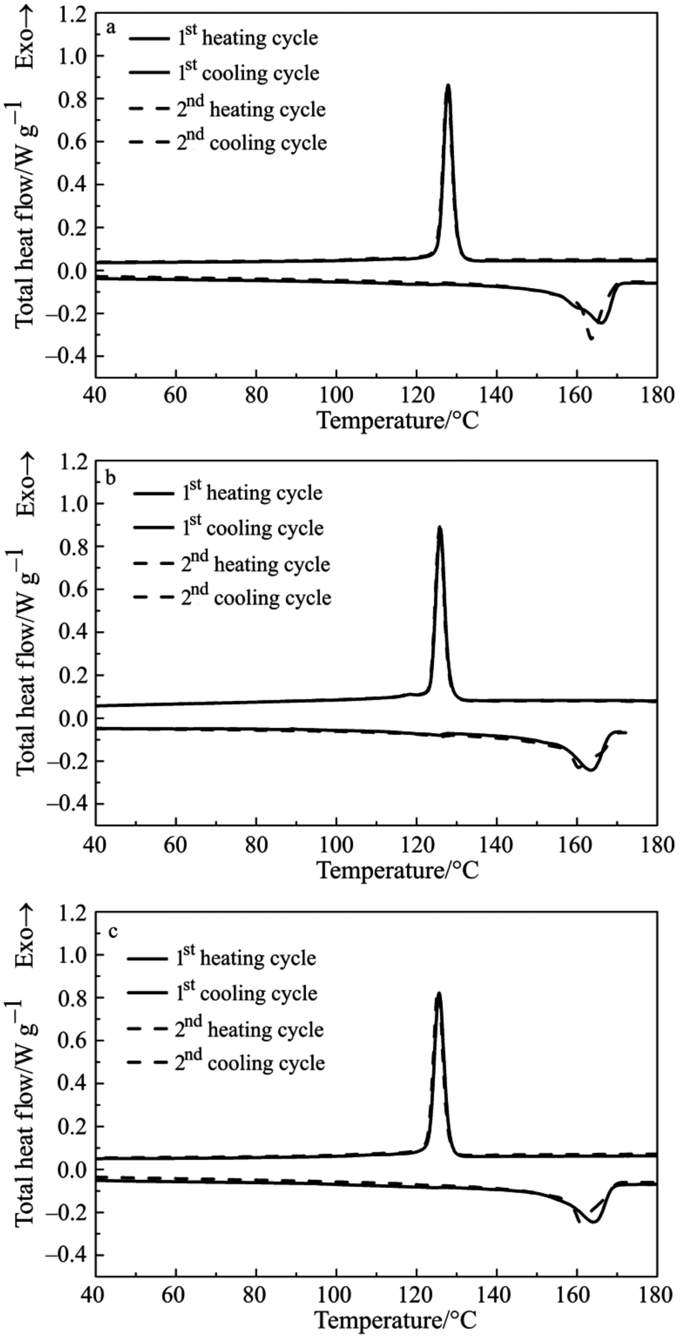

Fig. 3 Total heat flow curves for binary blends of PP PCW: a – I+M, b – I+B and c – M+B

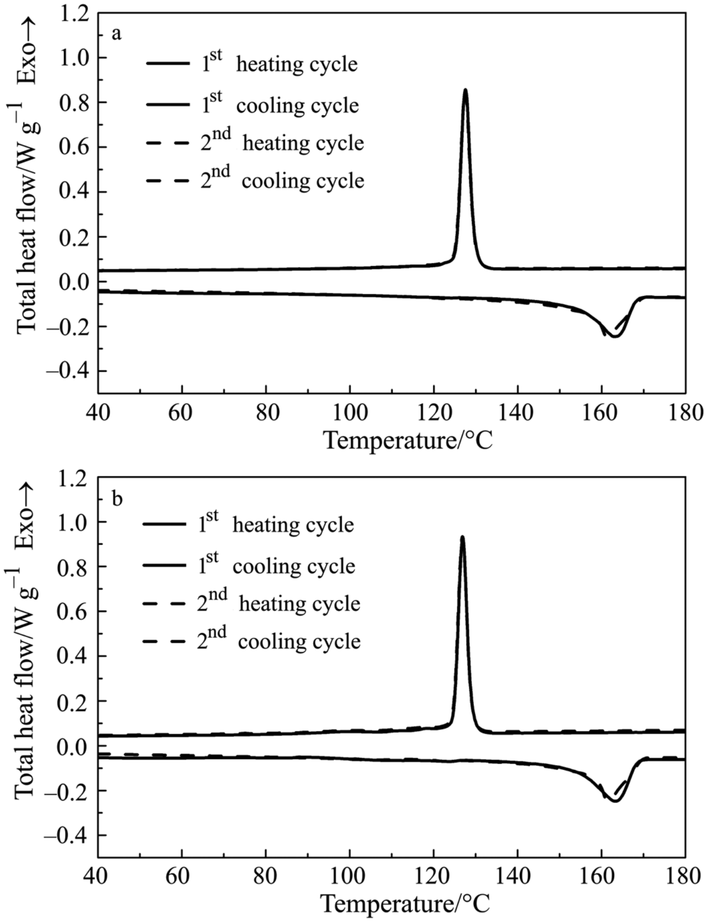

Fig. 4 Total heat flow curves for ternary blends of PP PCW: a – W0 and b – W10

Discussion

The PP PCW materials differ from virgin materials in that the PCW has been exposed to a number of processing cycles, i.e. during initial fabrication, during pelletisation of PCW flakes and during injection moulding into test bars. In addition, the PCW was also exposed to environmental degradation (UV radiation) during residence at the MRFs. In the present study, the above processes did not affect the crystalline structure of the PCW grades; both virgin and PCW materials crystallised as α-PP. This is different from other researchers [1, 2] who found that under severe processing conditions (e.g. mixing at high rpm) or under radiative conditions (e.g. prolonged UV radiation), PP could crystallise as β and/or γ phases. The change in heat capacity curves between the first and second heating cycle indicates that PP PCW are affected by thermal history of common processing conditions such as extrusion and injection moulding. How-

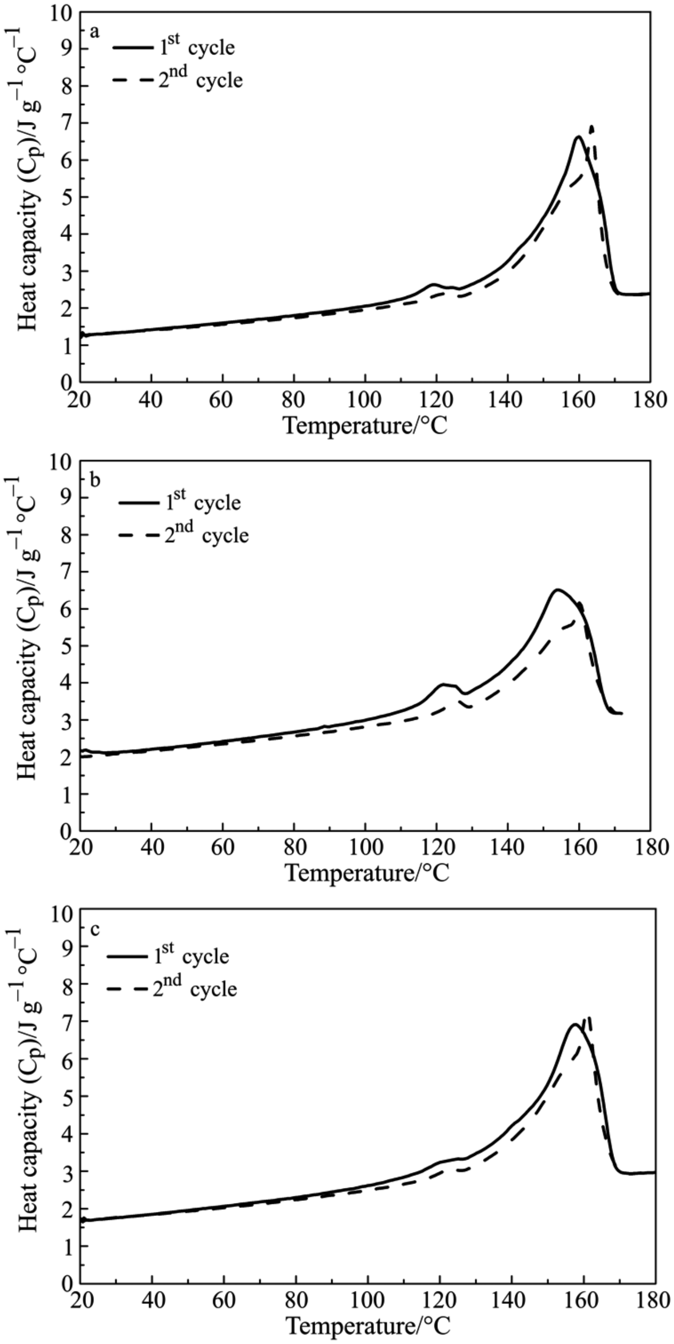

Fig. 5 Heat capacity curves for binary blends of PP PCW: a – I+M, b – I+B and c – M+B

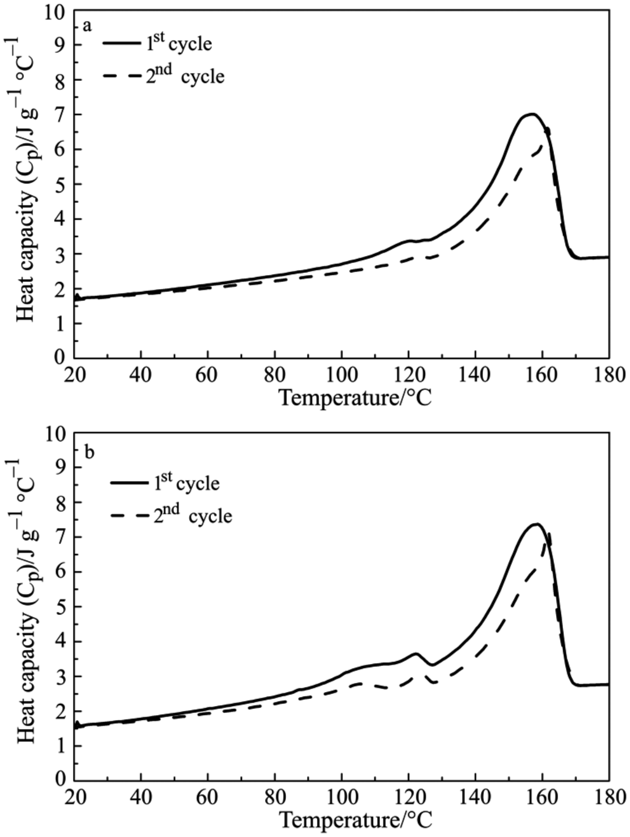

Fig. 6 Heat capacity curves for ternary blends of PP PCW: a – W0 and b – W10

ever, absence of any β or γ phases suggest that PP PCW can be processed using common manufacturing techniques without affecting the crystalline structure of PP.

The PP PCW products are collected together with other polymeric and non-polymeric products. As a result, the PP PCW materials are commingled with other materials. Processing of PP PCW therefore requires a rigorous sorting and cleaning process to remove these extraneous materials, and this was performed in the present study. However, the presence of PE peaks in the heat capacity curves corresponding to LLDPE and HDPE suggests that it is not possible to completely separate these polymeric contaminants from PP PCW.

The major sources of LDPE and LLDPE in the PCW stream are ice cream container lids and, bags and films respectively. In the present case, these were removed from PP during the manual sorting process. However, the fact that LLDPE was detected in the washed PP PCW indicates that fragments of bags and films were not removed completely. In this context it should be noted that the PP PCW materials in the present study were food packagings and, so, had food remnants in them. In the case of I and M, the food remnants caused sticking of small fragments of bags and films to the PCW and, as a result, complete removal of these fragments was not possible.

The minor peak temperature for B was, however, higher than the peaks observed in I and M. This suggests that a different type of PE, most likely HDPE, was present in B. This grade (B) was relatively free of food remnants, bag and film fragments although it contained fragments of labels which often have an HDPE coating [12]. It is likely that the observed PE peak in B is due to the presence of such labels. It is worth noting that this does not apply to I and M since direct printing is used to label these products.

A comparison of the enthalpies of the PCW materials with those for comparable virgin materials shows lower enthalpies for PCW materials (Tables 3 and 4). The difference is significant for all grades, and most significant for the high molecular mass (low MFI) PPs (M, B) than for low molecular mass (high MFI) I. As discussed earlier, PCW differs from virgin PP in that PCW may undergo degradation and may contain contaminants. The lower enthalpies of PCW grades therefore may be an effect of these factors.

The effect of a polymeric contaminant on enthalpy of PCW materials was specifically studied for ternary blends, with or without addition of 10% LDPE, W10 and W0. The results showed that the enthalpies of the two blends were similar. This suggests that the LDPE did not have a significant influence on the enthalpies of PP PCW. The quantity of PE in the individual PCW grades and binary blends is expected to be lower than 10%. Therefore the lower enthalpies of the PCW grades with respect to virgin PP grades are probably not due to PE contamination.

The PCW materials also contained organic and inorganic pigments, metal and glass pieces and dust and dirt particles [16]. If any of these contaminants acted as nucleating agent as claimed by Baquero *et al*. [13], the enthalpies of PCW would have been greater than that for virgin PP. Contrary to this, the enthalpies of PP PCW in the present study were lower than that for the virgin grades. The contaminants in PCW are present in a range of sizes and are of a variety of materials. It is possible not all of these are effective as nucleants for PP and, consequently, the presence of contaminants has not influenced the crystallisation significantly.

It is well known that PP readily undergoes chain scission due to thermal and mechanical loading and/or due to exposure to UV radiation [17–19]. In the present case, PP PCW materials had undergone chain scission as evidenced by higher MFI of these materials compared with virgin grades (Tables 1 and 2), i.e the molecular mass of the PCW grades were lower than the virgin PP grades. Reduction in molecular mass is a consequence of chain scission of PP under the influence of UV radiation, such as when left in sunlight at MRFs. The degradation of PP would also lead to the formation of chemical species such as hydroperoxide and carbonyl groups [2]. During crystallisation, the perfect chains crystallise, while the defective chains containing the chemical species remain in the amorphous phase. It may be argued that shorter chains, formed as a result of chain scission, enhance the crystallisation process due to their greater mobility. However, the growth of the crystals is also hindered by the presence of non-crystallisable defective chains ahead of the growth front. In the present case, the crystallisation process in PCW appears to be controlled more by the presence of defective chains than by the effect of shorter chains, resulting in the observed lower enthalpies in PCW compared with the virgin PP.

The fact that the difference in enthalpies is greater for the higher molecular mass grades (Tables 3 and 4), further suggests that enthalpies in PCW are affected by degradation. This is due to the fact that high molecular mass 'tails' present in high molecular mass PPs tend to undergo more chain scission under degradative conditions [19]. Such 'tails' are not present in low molecular mass PPs and therefore the extent of degradation in these grades is lower. Greater degradation in high molecular mass PPs reduces the number of perfect chains, which are capable of crystallisation, and consequently the enthalpies of these grades decrease to a greater extent than those for the lower molecular mass PCW.

The enthalpies of PP PCW blends in the present study could in general be predicted by a linear additivity rule i.e. the enthalpies of the blends were the weighted average of the enthalpies of blend components. The blend I+M, however, was an exception. For this blend, the measured enthalpies were greater than the predicted values by ~8%, Table 5. The fact that the other two blends containing either I or M i.e. I+B and M+B did not show any deviation from the linear additivity rule indicates that a positive interaction occurs only when I and M are blended. The mechanism for this improvement in enthalpy for I+M is not currently apparent.

The fact that the enthalpies of PP PCW blends in general are a weighted average of the enthalpies of blend components is of great importance from a recycling point of view. Since PP packaging consists of a wide variety of shapes, sizes and grades, and, the quantity of these can vary geographically and seasonally, recycling the packagings as blends would be more economical than recycling individual grades. It has been reported [20] that blends of different grades of PP PCW may yield resins with unacceptable properties. However, the results in the present study indicate that it is possible to recycle different grades of PP PCW together as blends with predictable thermal properties.

Conclusions

The present study shows that enthalpies of PP PCW are significantly lower than those for comparable virgin PP. This reduction in enthalpies is a consequence of degradation of these materials during residence at the MRFs. Under the influence of UV radiation the PP chains undergo chain scission, which forms chain defects. Presence of these defective chains reduces the number of perfect crystallisable PP chains in the materials and therefore the enthalpies of PP PCW are reduced compared with virgin PP.

The PP PCW materials contain polymeric contaminants such as LDPE, LLDPE and HDPE even after a rigorous sorting and cleaning process. The enthalpies of PP PCW, however, are not altered by low amounts (up to 10%) of polymeric contaminants. The crystalline structure of PP PCW grades and blends is also not altered by either degradation or contamination; these crystallise as α-PP, similar to their virgin counterparts.

The enthalpies of PP PCW blends are in general intermediate to that of the constituent grades. The enthalpies of the blends follow a simple linear additive rule. The possibility exists for designing PP PCW blends for specific purposes from PP PCW grades of known enthalpies.

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