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Effect of Impurity Content on Long Term Performance of Recycled HDPE For Structural Application

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Abstract. thermoplastic wastes are some of the major components of global municipal solid waste (MSW), especially because of the large volume and low cost of these materials. Since the plastic wastes may be obtained from several sources, they might have been exposed to different storage and processing conditions and may exhibit different behavior depending on the degradation level. The impurity removal is critical and has significant effect in durability of the final product. In that context, the incorporation of waste in materials for structural application demands a series of technical evaluation procedures that need to be assessed, especially when the materials undergo constant pressure. This work aims to characterize blends with polypropylene (PP), which is commonly found in recycled high density polyethylene (HDPE) and to evaluate the effect of its content in both short-term mechanical behavior and stress cracking, comparing these properties to the ones of pristine HDPE. The study was carried out by means of tensile and impact tests, melt flow index (MFI), and Un-notched Constant Ligament Stress Crack Test (UCLS).

Keywords: Recycled HDPE; Structural Application; Long-term Performance.
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INTRODUCTION

Recycled and waste thermoplastics are some of the major components of global municipal solid waste (MSW), especially because of the large volume and low cost of these materials. HDPE is an attractive material for structural applications such as geomembranes and pipes for water transportation due to its low weight, high corrosion resistance and low cost, and it can be a suitable substitute for both steel and concrete. In these applications, two are usually the main causes for the failure: inadequate welding or the existence of small internal defects that generate a slow growth of cracks in the material [1,2].

Since recycled plastics can be obtained from various sources, they may have been exposed to different storage and processing conditions, and may exhibit different performance depending on their level of degradation. In this context, post-consumer recycled plastics can have various colors, grades and contaminants, producing final products with a wide variety of properties. The major concern in this case is in relation to contaminants. In addition, the polymeric materials are subject to thermal, chemical, mechanical and biological degradation. Therefore, the incorporation of recycled materials into products that have structural function demands a series of technical evaluations regarding the product applicability [2,3].

In that context, failure times in years are associated with the mechanism of crack formation in several stages: cracking, evolution of the damage zone and propagation of the damage to the rest of the material. Several tests have been introduced in recent decades to measure this effect, and the most accepted by scientific and industrial communities are probably Pennsylvania Edge-Notch Tensile (PENT) [4] and FNCT (Full Notch Creep Tests) [5]. Since recycled HDPE resins tend to exhibit less resistance to stress cracking due to degradation from reprocessing and also due to contaminant inclusions, conducting tests under very aggressive conditions may make it impossible to effectively compare different resins due to the low time of failure. In this context, there are two standardized methods for evaluating resistance to stress cracking of polymers: the Notched Constant-Stress (NCLS) and NCTL (Notched Constant Tensile Load Test), which are standardized by ASTM F2136 [6] and ASTM D5397 [7], respectively. The main difference between the NCLS and NCTL tests is the applied load: for the NCTL, the loads are based on the percentages of the yield stress of the material to be tested. For NCLS, a constant load of 600 psi (4.14 MPa) is used. This is the load applied in the NCTL test if the yield stress is 4,000 psi (4.14 MPa) and the load used is 15% of the material flow stress [8].

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The UCLS was developed to evaluate the effects of contaminant particles commonly found in recycled HDPE, mainly from post-consumer materials. The main difference between the UCLS and NCLS tests is that the UCLS test specimens are larger in size than the NCLS assay in order to increase the amount of contaminant particles in the same test specimen and induce crack growth and propagation. Another important difference of the two methodologies is that in the NCLS test a notch is realized to concentrate tension in the material and to favor the cracking under tension. In addition, the specimens are immersed in a surfactant medium to accelerate the cracking propagation process and material failure [9].

This work aims to characterize the influence of polypropylene (PP) in recycled high density polyethylene (HDPE) and the effect of its content in both short-term mechanical behavior and stress cracking, comparing these properties to the ones of pure HDPE. The study was carried out by means of tensile and flexural tests, melt flow index (MFI), and Un-notched Constant Ligament Stress Crack Test (UCLS).

MATERIALS AND METHODS

In order to evaluate the PP content commonly present in recycled HDPE, samples of both post-consumer and post-industrial commercial HDPE blends, provided by RRC Plásticos Company and Tigre ADS, both located in Rio Claro, São Paulo, Brazil, were chemically characterized. The blends descriptions are presented in Table (1):

Table (1) – HDPE blends.

Blend	Source	Shape
R.1	Post-consumer	Flasks
R.2	Post-consumer	Grinded
R.3	Post-consumer	Pellets
R.4	Post-industrial	Pellets

After the PP content evaluation, a series of simulated recycled blends with different PP content were produced. The HDPE used in this work was Braskem GP 5550, which exhibit high molar mass and excellent mechanical properties and high resistance to stress cracking. Therefore, this is a resin that can be used in structural applications such as corrugated drainage systems. The PP used in this work was Braskem HP 500D, which is a homopolymer with low flow rate, high stiffness and high impact resistance. Both polymers have close melt flow index (MFI).

The PP was selected once it is one of the most common impurity in recycled HDPE. Many consumer goods are produced with both materials, with the HDPE in the body and the PP in the lid. Moreover, PP was detected in all the recycled blends in significant amounts. Nonetheless, further study is necessary to evaluate the effects of other impurities such as inorganic additives, as well the synergic effect between them. The PP content was evaluated according to ASTM D7399-08[10], using an Infrared Spectrophotometer (FTIR) Thermo Fisher Scientific, model Nicolet iS10. The samples were analyzed using ATR method with Germanium crystal, resolution of 4 cm^{-1} and 32 scans in 4000-650 cm^{-1} range. The produced blends are presented in Table 2:

Table (2) – Blends produced for performance evaluation

Blend	Weight percentage (wt.%)	
	GP 5550	HP 500D
S.1	100	0
S.2	98	2
S.3	96	4
S.4	94	6
S.5	92	8

The blends were produced using an AX Plastics "AX 16-DR" co-rotating twin screw extruder with L/D 40, available in Laboratory of Chemical Processes and Particle Technology of IPT. The conditions used to process the blends are described as following:

– temperature profiles: 140 °C / 170 °C/175 °C/180 °C/185 °C /185 °C/170 °C /165 °C / 160 °C;
– feed speed: 10 rpm at 12 rpm;
– rotation speed of the screw threads: 105 rpm.

After extrusion of the formulations shown in Table 2, the pelletized materials were injected into test specimens for further evaluation of tensile, flexural, and UCLS, using a Ray-Ran Test Sample Injection Molding Bench Injector

Press available in Laboratory of Chemical Processes and Particle Technology of IPT. The conditions of the injection process were: cylinder temperature of 210 °C and mold temperature of 90 °C. The flexural tests were carried out according to ASTM D790-17 [11]. The test samples were conditioned to (23 ± 2) °C and (50 ± 5)%.R.H, for 40 h. Test velocity was of 1.40 mm/min and distance between supports was of 52,48 mm. The tensile tests were carried out using ASTM D638:-2014 [12]. The samples were conditioned at (23 ± 2) °C and (50 ± 5)%.R.H, for 40 hours. The test velocity was 50 mm/min using Type I test specimens. Both tensile and flexural tests were conducted using a Universal testing Machine, Emic, model DL10000. The Un-notched, Constant Ligament Stress Crack Test (UCLS) were carried out according to ASTM F3181-16. The specimens were produced based on ASTM D638 -Type I. The applied stress was 4.48 MPa and the temperature was of 80 °C. All the tests described above were conducted in Laboratory of Chemical Analysis of IPT.

RESULTS AND DISCUSSION

The Table (3) presents the results of PP content in the recycled HDPE blends. The results show that all samples exhibit PP content above 5wt.%, which could induce failure due to the HDPE/PP poor interphase. In that context, it is very important to assess the impact of PP content in both short and long-term performance of recycled HDPE.

Table (3) – PP content in the recycled HDPE blends.

Blends	**(1465 cm^{-1})**				**(1373 cm^{-1})**				**1373/1465 ratio**	**PP (wt.%)**
	A	**B**	**C**	**Average**	**A**	**B**	**C**	**Average**		
R.1	0,0193	0,0171	0,0195	0,0186	0,0016	0,0017	0,0020	0,0018	0,0948	6,1
R.2	0,0175	0,0115	0,0163	0,0151	0,0014	0,0013	0,0013	0,0013	0,0883	5,3
R.3	0,0210	0,0204	0,0205	0,0206	0,0019	0,0021	0,0016	0,0019	0,0905	5,6
R.4	0,0212	0,0192	0,0170	0,0191	0,0021	0,0021	0,0020	0,0021	0,1080	7,7

Table (4) shows the results of tensile, flexural and UCLS tests in the blends. In order to evaluate if the differences between the samples are significant, a single-step multiple comparison statistical test by Tukey Method was conducted. This method can be used on raw data or in conjunction with an ANOVA (post-hoc analysis) to find if means that are significantly different from each other in a significance level. For this work, the calculus were carried out using a level of 0.05, which represents the 95% confidence level, using the software Origin Pro 9.1.0 b215
(OriginLab Corporation). The results are presented in Table (5), on which signal equals 1 indicates that the difference of the means is significant at the 0.05 level and signal equals 0 indicates that the difference between the means is not significant at the 0.05 level. The results show that, for short-term performance (tensile and flexure tests), the means are significantly different only when comparing the maximum flexural strength of the S.1 with S.3 and S.1 with S.5. Nonetheless, in both cases the difference between the means corresponds to around 10 and 11%, respectively, which should not represent an increase of short-term performance with the increase of PP content. Nonetheless, it is known that due to the differences between the melt temperatures of the two polymers, the blend becomes immiscible and has poor physical and mechanical properties [3]. For this reason, it is often necessary to use compatibilizing agents to process these two polymers [13]. And, it is very important to evaluate the PP content in recycled HDPE, even when the short-term performance is adequate. Moreover, when the material is applied under constant stress in structural applications, it is necessary evaluate the long-term performance as well. This evaluation was carried out by UCLS tests. The results show a tendency of reducing failure time with the PP content is increased. The Turkey analysis showed that samples with low PP content (up to 4wt.%) do not present reduction in the time-to-failure, therefore a reduction of service life is not expected. The samples with 6% and 8 wt.% of PP exhibited a significant reduction in the time-to-failure, of around 30% and 70% respectively, which shows that the service life of this materials should be reduced, possibly prohibiting its application.

Table (4) – Tensile and flexural tests

Blend	Test sample	Tensile Tests		Flexural Tests		UCLS
		Yield strength (MPa)	Elongation (%)	Yield Strength (MPa)	Flexural Modulus (MPa)	Failure Time (Hours)
S.1	1	30.41	9.65	21.13	714	332
	2	29.75	9.09	21.05	801	321
	3	28.31	8.61	21.42	774	310
	4	30.37	9.05	21.41	754	340
	5	32.45	12.15	21.16	828	274
Avg.		30 ± 2	10 ± 2	21.2 ± 0.2	774 ± 54	315 ± 32
S.2	1	31.41	17.26	21.56	751	387
	2	31.51	9.87	21.36	784	318
	3	32.19	8.71	21.69	740	342
	4	30.77	14.20	22.00	719	305
	5	31.71	10.00	23.05	805	310
Avg		31.5 ± 0.6	12 ± 4	21.9 ± 0.8	760 ± 43	332 ± 42
S.3	1	29.45	8.67	22.62	713	274
	2	30.00	8.72	23.53	823	310
	3	25.69	12.37	23.39	783	240
	4	28.54	10.26	23.92	963	239
	5	27.75	11.22	22.06	831	290
Avg.		28 ± 2	10 ± 3	23.1 ± 0.9	823 ± 113	271 ± 39
S.4	1	27.16	12.99	23.93	777	220
	2	29.22	11.41	23.01	735	231
	3	32.17	13.45	22.22	835	250
	4	27.63	15.62	20.18	933	197
	5	29.04	11.15	21.27	736	210
Avg.		29 ± 2	13 ± 2	22 ± 2	803 ± 103	221 ± 25
S.5	1	28.98	11.55	23.70	847	138
	2	27.27	14.29	22.81	802	97
	3	29.49	14.42	23.66	796	110
	4	33.74	9.42	23.07	919	121
	5	32.17	8.03	23.91	859	111
Avg.		30 ± 3	12 ± 4	23.4 ± 0.6	845 ± 61	115 ± 19

Table (5) – Multiple comparison by Tukey Method

Comparison	Tensile		Flexure		UCLS
	Yield strength	Elongation	Yield Strength	Flexural Modulus	Time-to-failure
S.2-S.1	0	0	0	0	0
S.3-S.1	0	0	1	1	0
S.3-S.2	0	0	0	0	1
S.4-S.1	0	0	0	0	1
S.4-S.2	0	0	0	0	1
S.4-S.3	0	0	0	0	0
S.5-S.1	0	0	1	1	1
S.5-S.2	0	0	0	0	1
S.5-S.3	0	0	0	0	1
S.5-S.4	0	0	0	0	1

CONCLUSIONS

The chemical analysis of recycled HDPE blends showed that all samples exhibited PP content above 5wt.%, which could induce failure due to the HDPE/PP poor interphase. The tensile and flexure tests comparison between the pure HDPE and the blends with PP presented a maximum variation of around 11%, which should not represent an actual change in the short-term performance when the PP content is increased. Nonetheless, the results of UCLS tests showed a tendency of reducing failure time when the PP content was increased. The samples with 6% and 8wt.% of PP exhibited a significant reduction in the time-to-failure, of around 30% and 70% respectively. Therefore, the service life of these materials could be reduced significantly, possibly prohibiting their application.

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