



# Article Effect of the Intercalation and Dispersion of Organoclays on Energy Demand in the Extrusion of Recycled HDPE/PP Nanocomposites

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Abstract: Few studies have drawn on any systematic research into the energy demand to produce polymer-based nanocomposites. Regarding the problem, it is well-known that single screw extrusion is an energy-intensive process, so the incorporation of energy meters must be considered to examine the energy efficiency of the process. In this study, the effect of a nanoclay addition on the energy demand of the extrusion process was examined by extruding recycled high-density polyethylene (rHDPE) and recycled polypropylene (rPP) with a gradual compression screw with both dispersive and distributive mixers. The rHDPE/rPP was modified by adding commercial organoclay (OMMT) (3 wt%) and olefin block copolymer (OBC) (5 wt%) as compatibilizers. The energy consumption was measured on the total energy of the extruder machine. Mass throughput (MT) and specific energy consumption (SEC) were obtained at different screw speeds (10, 20, 30, 40, 50 RPM). The SEC of OMMT and OMMT/OBC nanocomposites was 25-50% lower than rHDPE/rPP, especially at higher throughputs. X-ray diffraction (XRD) and scanning electron microscope (SEM) illustrated the degree of intercalation and dispersion of the organoclay at different screw speeds. Better organoclay intercalation and dispersion were found at lower temperatures. Rheological curves showed a decrease in the viscosity at extrusion rates of nanocomposite mixtures. Melt temperature measured at die exit was reduced in the presence of organoclay over the screw speeds studied. This work suggests that the processing of rHDPE/rPP based nanocomposites can result in minor costs when processing conditions are carefully selected.

Keywords: recycled plastics; energy demand; polymer nanocomposites

# 1. Introduction

Nowadays, plastic recycling is one of the keys to preserving our planet's resources, as the World Economic Forum (WEF) [1] encourages. Additionally, it can remarkably transform the global market of the plastics packing industry in line with the circular economy. It is estimated that recycling plastic produces a lower carbon footprint associated with emissions than the production of raw plastic materials [2]. Furthermore, mechanical recycling has several advantages for post-consumer plastics. These include relatively simple processes, appropriate for most plastics, low pollution risk, and environmental suitability in transition to the circular economy [2]. Therefore, recycling can radically contribute to this new economy in several areas in the plastics markets. The New Plastics Commitment of the WEF promotes increasing the use of recycled plastics and their quality to make durable



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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). products by 2025 [1]. This has recently boosted several studies on improving the miscibility, quality, and processability of mixed plastics to develop industrial products [3,4].

Polyethylene (PE) and polypropylene (PP) represent more than 50% of the major thermoplastics' total global demand distribution for 2020 [5]. Similar trends are illustrated in plastic consumption in Ecuador, as shown in Figure 1. In addition, plastic solid waste (PSW) comprises mainly PE and PP, which is traditionally used in the packing industry in Ecuador [6]. The increase in plastic recycling must counteract the consumption of plastics. Local regulations encourage using recycled plastics in the products, so plastic recycling plastics. However, mixing plastics to develop feasible materials to manufacture high-quality plastics is still a challenge [7]. Thus, it is relevant to Ecuador to develop a methodology to determine the relative concentrations and characterization of PE/PP mixtures [8] and decide on appropriate strategies to upgrade these mixtures on an industrial scale. Thus, the value of recycled plastics should be radically improved to make plastic materials more economical, with better performance, and are environmentally sustainable.



Figure 1. Distribution of primary plastics consumption in Ecuador for 2019 [9].

Typically, some additives have been used to improve the miscibility between PE and PP, such as ethylene vinyl acetate (EVA), ethylene propylene diene rubber (EPDM), and styrene ethylene butylene styrene (SEBS) [10]. Generally, compatibilizer additives can be expensive, but recycling plastics can still be economical with these additions at 2–5 wt% [10]. Organoclays have been used in the past to improve plastic miscibility and develop new materials with high performance. Recently, the literature has shown an improvement in the physical properties and processability in recycling plastics incorporating organoclays [11,12]. Thus, upgrading recycled plastics using organoclays or nanoclays can generate value-added waste plastics. We have successfully processed immiscible recycled plastic nanocomposites using a single-screw extruder incorporating dispersive and distributives mixers [8,13]. Despite all benefits, limited attention has been observed in the energy demand in processing recycled plastics incorporating organoclays or other compatibilizers.

The effect of the intercalation or exfoliation of the organoclays in the polymer extrusion process has been extensively studied [14–16]. The degree of intercalation and dispersion is affected by the organoclay chemistry [17] and the screw design [18,19] in the polymer extrusion process. The extrusion process is the most widely used technology for plastic product manufacturing in Ecuador [20]. Some modifications in the extrusion machine such as appropriate screw design to increase the shear intensity are required to develop

a nanocomposite structure. X-ray diffraction (XRD) data allow for the determination of the nanocomposite structure [21], while the degree of dispersion has been successfully observed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) [22]. Therefore, the nanocomposite structure and organoclay dispersion have been studied to correlate with the organoclays or polymer chemistry, capability of dispersion, and processing extrusion conditions.

Although plastic processing is lower in energy demand than other materials, it can be expected that energy demand for the recycled plastics process could be an inefficient energy process. The specific energy consumption (SEC) in polymer extrusion has been studied to correlate polymer materials and processing parameters [23]. The foremost vital parameters considered in the extrusion machines are the speed of the extruder screw rotation, the temperature in all the heaters, and the pressure developed by the extruder. However, the drive motor of the extruder is the main examination for energy efficiency in the extrusion process [24]. The SEC is reduced in polymer extrusion as screw speed increases in a single screw machine, despite the difference in the screw geometry design [22,24,25]. This reduction is due to the viscous shear or melt viscosity generation, which is excellent at medium or higher screw speeds. Nevertheless, the mass throughput (MT) differs between single and barrier flighted screws [26]. For instance, higher MT in the barrier screw was correlated to higher processing temperatures due to higher free volume than a singleflighted or conventional screw, and reduced viscosity improved the MT as it increased the processing temperature. Moreover, the different configuration of a single screw is also correlated to the mixer drive power, melt pressure, and melt temperature rise through each mixer [27]. Wong et al. [26] also illustrated that Maddock or Pineapple mixers included in a conventional screw increased the MT and decreased the resident time compared to a traditional or single flighted screw; these mixers have an influence on obtaining earlier completion of the melting process, then the degree of mixing was enhanced compared to the barrier or singled flighted screw.

Regarding energy demand in polymer extrusion studies, Vera-Sorroche et al. [23] illustrated that a higher molecular weight of high-density polyethylene (HDPE) would lead to a higher MT than a lower molecular weight HDPE using a tapered screw despite the processing temperature. In contrast, the lower molecular weight of HDPE resulted in higher extruder throughput in barrier flighted screws at high temperature processing. Higher values of MT have been correlated to polymer densities. Higher density for PS would lead to lower SEC than HDPE in different screws. Therefore, intrinsic properties such as viscosity or frictional characteristics would lead to an effect on drag flow or thermal conductivity, leading to differences in MT or SEC [28]. In [24], it was demonstrated that SEC depends on two critical conditions such as screw speed and die restriction. For these reasons, energy efficiency is linked to the screw geometry, polymer properties, processing temperatures, or screws speed [23,28]. Therefore, the challenge is to optimize the energy consumption and keep the melt quality during the extrusion process.

Generally, the studies have been related to extruding virgin polymers and different screw types in a single screw process by varying the processing conditions to determine their effect on the melt quality and energy consumption. However, there is no correlation between using recycled plastics and upgrading to understand potential optimization in energy demand by using fillers or compatibilizers. In addition, this research used an industrial scale machine to understand the potential application to the recycled plastics industry. Therefore, recycled high-density polyethylene (rHDPE)/recycled polypropylene (rPP) was modified by an organoclay and olefin block copolymer (OBC) as compatibilizers. The intercalation and dispersion of the organoclay and its effects on the energy demand were studied by processing rHDPE/rPP in a single screw machine. The screw was tapered compression geometry incorporating Maddock, Rings, and Pineapple mixers. A noninvasive thermal imager obtained the composite melt temperature at the end of the extruder at different screw speeds.

## 2. Materials and Methods

## 2.1. Materials

Recycled high density polyethylene (rHDPE)/recycled polypropylene (rPP) was donated by INTERCIA S.A., Guayaquil, Ecuador. An experimental D5541 olefin block copolymer (OBC) (Dow Chemical) was supplied by Entec Polymers, Orlando, FL, USA. This copolymer has a density of 0.863 g/cm<sup>3</sup>, a melt flow index of 6.5 g/10 min, and a melting point of 190 °C. The organoclay, Cloisite 20A, with a CEC = 95 meq/100 g-clay and  $d_{001}$  = 24.2 Å was obtained from Southern Clay Products, Gonzales, TX, USA.

### 2.2. Preparation of Nanocomposites

The recycled plastics and organoclays/compatibilizer were dried at 60 °C for 24 h using a Microprocessor Drying Oven—Q317M-33 from QUIMIS, Sao Paulo, Brazil. All formulations were made of rHDPE/rPP with the addition of the OBC compatibilizer and Cloisite 20A at 5 wt% and 3 wt%, respectively, as shown in Table 1. Melt blending was performed using a single screw extruder with a ratio of L/D = 26 and a screw diameter of 65 mm. The metering zone in the screw incorporated Maddock, Rings, and Pineapple mixers, as illustrated in Figure 2. Thus, low and high shear processes were used to balance the mixing of the organoclay and compatibilizer in recycled polymers using this modified single screw. The masterbatch of recycled plastic composites was extruded following a temperature profile at 190/240/270/250/220 °C (from the feed end to die end) and a screw speed from 10 to 50 rpm in steps of 10 rpm. The process was stabilized for about 30 min at each screw speed. Samples were shaped using steel mold by compression molding for characterization studies.

Table 1. Recycled plastic compositions.

Blend	Description
1	rHDPE/rPP
2	rHDPE/rPP/5OBC
3	rHDPE/rPP/3C20A
4	rHDPE/rPP/3C20A/5OBC



**Figure 2.** From the end of the screw: (**a**) dispersive mixer (Maddock) and distributive mixers (**b**) Rings, and (**c**) Pineapple in the screw design.

## 2.3. Characterization and Measurement

X-ray diffraction measurements were obtained using a Phillips PANalytical XPert-Pro model diffractometer equipped with Cu K $\alpha$  ( $\lambda$  = 1.54060 Å) radiation operated at 40 kV and 30 mA with a 1/8" divergent slit and 1/16" anti-scatter slit, scanning range 1° to 40° (1 $\theta$ ), 0.05° step size, 20 s per step.

Micrographs were obtained using an FEI Inspect S50 scanning electron microscope (SEM) in low vacuum mode with 15–20 Pa of pressure in the sample chamber. Micrographs were taken using the backscattering detector (BSED) operating with an acceleration voltage of 4–4.5 kV and a spot of 4–4.5. The size and distribution of the clay agglomerates were characterized using image analysis software (ImageJ from Fiji, developed by University of Wisconsin-Madison) that enabled the calculation of the area of clay agglomerates.

Rheological properties were tested using a Malvern Kinexus Pro+ Rotational Rheometer at 180  $^{\circ}$ C consisting of parallel plates with a 1.85 mm gap setting; shear rate varied

between 0.1 and  $100 \text{ s}^{-1}$ . The rheological curves were smoothed using Origin 6.0 software. For crystallinity and concentration of the recycled plastics, the enthalpy of fusion was obtained by dynamic scanning calorimetry (DSC), model Q200, TA Instruments; the procedure for both analyses can be found elsewhere [29]. The total energy consumption of the extruder was measured using a Fluke 435, as observed in Figure 3a. All energy measurements include the motor, heaters, and cooling fans. A thermal imager, TESTO 885, was used to examine the melt temperature at the end of the extruder die. This allows one to measure the melt temperature homogeneity, as observed in Figure 3b,c.



Figure 3. An energy analyzer (a) and thermal imager (b,c).

## 3. Results and Discussion

#### 3.1. Studies of Nanocomposite Morphology

Figures 4 and 5 illustrate the effect of the organoclay and the OBC in the structure of the nanocomposites at different screw speeds using XRD diffraction. XRD technique has been used to analyze surfactant expansion of the spacing of clay interlayers. Cloisite 20A (C20A) has primary reflections at about 3.54° (~25 Å). C20A has shown a feasible dispersion in either HDPE or PP in the extrusion process [13,17]. In this research, the C20A nanocomposites showed a primary  $(d_{001})$  and secondary diffraction  $(d_{002})$  peak at about 2.2–2.67° (32.9–39.5 Å) and 6.54–6.85° (12.9–13.5 Å), respectively. The additional extrusion processing to prepare the samples via compression could lead to decreases in the basal spacing of the stacks. The polymer chains were able to intercalate in the clay galleries for all cases since the d<sub>001</sub> of C20A was shifted to greater basal spacing. There was no significant effect of the OBC in the intercalated nanocomposite structure. Figure 6 illustrates the corresponding interlamellar distances of the clay layers in the mix of rHDPE/rPP all screw speeds. The greater d-spacing (39.5 Å) was observed in C20A nanocomposite at 10 RPM. In comparison, the lowest d-spacing (33 Å) at 50 RPM was observed in the C20A/OBC nanocomposite. There is low residence time at higher speeds; thus the grade of intercalation decreased despite the presence of OBC. After 20 RPM, the C20A/OBC nanocomposites had great d-spacing compared to their counterparts at a similar speed. The OBC effectively improved the miscibility between rHDPE and rPP [8]. However, OBC does not significantly affect clay interaction due to its low capacity to promote intermolecular interaction between the surfactant of the clay and polymer chains. Therefore, there should be a balance in the system's chemistry, shear stress in melting the polymer, and appropriate residence times in the single screw extrusion process.



**Figure 4.** X-ray diffractometry of the 97% rHDPE/rPP and 3% C20A blend at different screw speeds, JCPDS-Card No. #00-007-0330.



**Figure 5.** X-ray diffractometry of the 92% rHDPE/rPP, 3% C20A, and 5% OBC blend at different screw speeds, JCPDS-Card No. #00-007-0330.



Figure 6. Interlamellar distance d001 of the 97% rHDPE/rPP and 3% C20A blend at different screw speeds.

Dispersion of the clay in the polymer matrix rHDPE/rPP at different screw speeds was characterized via scanning electron microscopy (SEM). Figures 7 and 8 show the morphology obtained for the samples with and without the OBC compatibilizer. Tactoids were observed in all nanocomposites. C20A dispersion quality was improved by reducing the presence of larger agglomerates with OBC as the screw speed increased. However, C20A showed higher clay agglomerates as the screw speed increased. As observed previously, the intercalation process can be seen to have two steps: first, the stacks of platelets are decreased in height; and second, the polymer chains diffuse into the galleries. The exit die temperatures of the C20A and C20A/OBC nanocomposites changed from 196 °C (10 RPM) to 231 °C (50 RPM) and 220 °C (10 RPM) to 244 °C 50 RPM), respectively, as shown in Table 2. Thermal degradation of the surfactant in C20A was observed after 220 °C by Normand et al. [18].

Thus, the decrease in the intercalation was due to thermal degradation of the surfactant due to higher temperatures and viscous dissipation, usually developed at higher screw speeds. This is why C20A/OBC showed a lower average area than the C20A counterpart at each screw speed, as illustrated in Table 2. Despite the surfactant degradation, a similar average area was observed in the organoclay nanocomposite basal spacing at 40 and 50 RPM due to the compatibilizer's effect, which improves the dispersion of the clay in these recycled plastics. Therefore, the compatibilizer plays a relevant role forming bigger particles due to physical interaction with the clay. As a result, the presence of the surfactant increases the interaction between the organoclay and the rHDPE/rPP matrix and the shear development must be balanced due to screw geometry and processing conditions to improve the dispersion in the organoclay nanocomposite extrusion.



**Figure 7.** SEM images of the 97% rHDPE/rPP and 3% C20A blend at different screw speeds (a) 10 RPM, (b) 20 RPM, (c) 30 RPMm, (d) 40 RPM, (e) 50 RPM.



**Figure 8.** SEM images of the 92% rHDPE/rPP, 3% C20A, and 5% OBC blend: (**a**) 10 RPM, (**b**) 20 RPM, (**c**) 30 RPM, (**d**) 40 RPM, and (**e**) 50 RPM.

**Table 2.** Average area of Cloisite 20A dispersed in rHDPE/rPP matrix with/without the OBC compatibilizer.

		10 RPM	20 RPM	30 RPM	40 RPM	50 RPM
rHDPE/rPP/C20A	Average area [µm] <sup>2</sup>	$22.3\pm0$	$31.0\pm5.7$	$35.6\pm16.0$	$44.0\pm5.5$	$45.6\pm7.7$
	Die temperature [°C]	196	212	225	227	231
rHDPE/rPP/C20A/OBC	Average area [µm] <sup>2</sup>	$119.7\pm24.6$	$67.6\pm5.8$	$63.2\pm16.2$	$45.2\pm3.8$	$41.6\pm7.5$
	Die temperature [°C]	220	229	234	240	244

# 3.2. Thermal Studies

DSC thermograms of recycled samples showed endothermic peaks from melting temperature of high-density polyethylene (HDPE) and polypropylene (PP) at about 134 °C and 163 °C, respectively. The endothermic peaks of PP were small and from quantification analysis via DSC, a polypropylene that was observed in composition at about ~2% by a methodology previously used [13]. The percent of crystallinity ( $%X_c$ ) and melting temperature (T<sub>m</sub>) of the rHDPE/rPP composites were obtained by DSC analysis, as shown in Table 3. The rHDPE had higher crystallinity than rPP due to its linear chain and lower branching compared to rPP. The compatibilizer OBC and C20A did not influence the crystallinity of the rHDPE/rPP samples; however, rPP showed a more significant crystallinity reduction than rHDPE in the presence of C20A or OBC/C20A. This reduction could be attributed to C20A, which has better compatibility with rPP in the recycle blend. The C20A showed an intercalated morphology in these polymers and no effect on the melting temperature of both plastics. This study confirms that the melting temperature and crystallinity of these mixed recycled plastics were not significantly affected by organoclay.

Description	rHD	DPE	rP	Р
	T <sub>m</sub> (°C)	X <sub>c</sub> (%)	$T_m$ (°C)	X <sub>c</sub> (%)
rHDPE/rPP	134.2	56.9	162.7	1.3
rHDPE/rPP/5OBC	134.2	56.7	162.7	1.2
rHDPE/rPP/3C20A	134.1	56.5	162.6	0.7
rHDPE/rPP/3C20A/5OBC	134.3	52.6	161.9	0.7

**Table 3.** Melting temperature  $(T^m)$  and percentage of crystallinity  $(\% X_c)$  of the rHDPE/rPP composites.

#### 3.3. Rheological Studies

Figure 9a illustrates the comparison of shear viscosity as a function of shear rate for all samples. The viscosity at the Newtonian region of rHDPE/rPP was lower than those systems with the organoclay. The effect of the reduced viscosity is related to dispersion and interaction between the C20A and the rHDPE/rPP. In contrast, the viscosity decreased with the addition of C20A due to the alignment of the platelets from organoclay that is parallel to the flow, as expected in plate-plate experiments and higher shear rates in the extrusion process. It can be inferred that intercalate organoclay recycle nanocomposites were high shear sensitive in the extruder region in rHDPE/rPP/C20A. The presence of OBC decreased the shear viscosity compared to un/additive recycled plastic composites at lower shear rates. Based on these results, the OBC and OBC/C20A appeared to improve the rHDPE/rPP clay nanocomposite processability at the extrusion region. Figure 9b displays the changes in the melt temperature at the exit die for all recycled composites. The rHDPE/rPP and rHDPE/rPP/OBC showed the highest melting temperature compared to other systems. In general, the mixing elements usually reduced the temperature variability in plastics, as observed by other authors [29]. The decrement in systems of C20A was possible due to a reduction in viscosity dissipation of the intercalated organoclay in the polymer matrix. Organoclays has been shown to improve the thermal stability of this polyolefin-based nanocomposite [30]. However, OBC has the lowest temperature variation from 10 to 50 pm due to improved thermal homogeneity in the system. The C20A and C20A/OBC increased the melting temperature deviation profile from low to high volume throughput (VT). The interaction between the surfactant and the polymer is purely enthalpic, reducing the friction between the nanoparticles and the polymer and influencing the viscous dissipation values.



**Figure 9.** Shear viscosity measured using a plate-plate rheometer at 180  $^{\circ}$ C (**a**) and melt temperature as a function of volume throughput (**b**).

Figure 10a compared the extruder's volume throughput (VT) from the recycled composites over different processing conditions. As expected, the screw speed was directly correlated to the throughput, but this screw design improved the throughput of the extruder with the addition of organoclay/OBC. The VT is deeply influenced by the thermal and viscosity and frictional properties of the polymer/filler and the screw design of the extruder [27], as observed in a single screw modified with Maddock and Pineapple mixers [26,27]. Hence, the VT of rHDPE/rPP–clay nanocomposites showed a better performance at higher shear rates.



Figure 10. Volume throughput (a), energy (b), and specific energy (c) as a function of screw speed.

Figure 10b,c illustrates the extruder's energy and specific energy consumption (SEC) including the motor and the fans at different screw speeds for all samples. As expected, the energy developed for the addition of C20A increased as the screw speed increased, with higher values at 40 and 50 rpm. Thus, high mixing shear is correlated to the energy capacity to develop at great torque in this extrusion process. However, due to higher VT, the value of SEC decreases in the nanocomposites. rHDPE/rPP/C20A/OBC showed the lowest SEC compared to other samples and the highest SEC was observed in rHDPE/rPP. As reported before by other authors, there is a trend in decreasing the SEC values as screw speed increased in the process of polyolefins [29]. The lowest values of SEC at higher screw speeds were correlated with the lowest values of shear viscosity at higher shear rates in the system with C20A. A similar correlation between SEC and shear viscosity has also been observed in polymer extrusion [31]. Thus, an appropriate selection of geometry screw, compatibilizer, and processing conditions should be performed to improve the consumption energy in single screw extrusion processes.

# 4. Conclusions

The addition of organoclays or compatibilizers to recycle plastics influences the values of shear viscosity, melt temperature, volume throughput, and specific energy consumption in a single modified screw process. The melt temperature profiles in recycled plastic nanocomposites were lower than those in recycled plastics without clay. Olefin block copolymers (OBC) reduced the temperature variation at different screw speeds. The VT was enhanced by adding C20A and OBC in rHDPE/rPP composites, but C20A increased the energy considerably at higher speeds. Moreover, the SEC was decreased in C20A systems compared to other composites. In this study, SEC values were very close at higher shear rates, so the extrusion must operate at higher shear rates in recycled clay nanocomposites. XRD and SEM results showed the intercalation and dispersion of the organoclay recycled plastics process in the extruder machine using a modified screw. The surfactant of the organoclay suffered degradation at most screw speeds in this recycled clay nanocomposite. The addition of the compatibilizer was beneficial for the physical interaction reduction between the particles and the screw; however, reducing the clay agglomerate area was not valuable. The compatibilizer OBC did not improve the interlamellar distance but counterbalanced the thermal degradation of the surfactant to the clay, achieving a decent organoclay dispersion. OBC improves processability as the screw speed increases. The nanoclay was beneficial for the polymer matrix regarding thermal stability and great SEC compared to a neat rHDPE/rPP mixture.

# 5. Future Work

Future work concerns the physical and energy characterization of the recycled composites by using other inexpensive fillers such as agricultural or industrial waste (e.g., rice husk or brewer spent grain). To determine the effect of the processing in developing different morphology structures and its correlation in energy consumption for the single screw extrusion process, the melt temperature and shear viscosity will be correlated to the specific energy consumption. Studies will also analyze the use of a different die to obtain materials directly to perform mechanical properties and determine the potential to manufacture products with low energy consumption and environmental consciousness.

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