



Research paper

Peelable clay/PE nanocomposite seals with ultra-wide peelable heat seal temperature window



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ABSTRACT

This study shows that the controlled dispersion and distribution of nanoclay in clay/polyethylene (PE) nanocomposites result in peelable sealants with an ultra-wide peelable heat-seal temperature window (ΔT_p). Different nanoclays, unmodified montmorillonite (Mt) and organo-modified Mt. (OMt), were examined for their capacity to generate peelable clay/PE nanocomposites in a melt-blending film extrusion process. Clay/PE nanocomposite films containing 6 mass% organoclay (OC) exhibited a cohesive peel behavior with a ΔT_p of about 12°C while PE film containing 6 mass% unmodified clay resulted in microcomposite with a lock seal performance. With the same mass% of OC, a much lower peel strength with a significantly broad ΔT_p of 45°C was achieved mainly due to the fine dispersion of OMT when PE-grafted-maleic anhydride (PE-g-MA) was used as a compatibilizer. WAXD and TEM results confirmed that a substantial enhancement in the dispersion and distribution of intercalated nanoclays was achieved with PE-g-MA compatibilizer. Finally, an ultra-wide ΔT_p of over 100°C was obtained for compatibilized OC/PE nanocomposite sealant containing only 3 wt% OC with partially exfoliated microstructure indicating that the exfoliation of nanoclay is in favor of peelability. The peel fracture mechanics of the clay/PE nanocomposite sealants were examined through T-peel test and SEM imaging. The results showed that the dispersion and distribution of nanoclay along with the interfacial adhesion between nanoclay and matrix are the key factors controlling the peel performance of the sealants.

1. Introduction

Packaging with peelable functionality is increasingly growing due to modern life style and is an important factor in consumer purchase behavior (Yoxall et al., 2006; Ahmed et al., 2014). Generally, peelable sealants are classified into adhesive peel (Ernesto Mendoza-Navarro et al., 2013; Nase et al., 2014), cohesive peel (Nase et al., 2008b; Liewchirakorn et al., 2017) or a mixture of both (Martínez-García et al., 2008). In contrast to adhesive peelable sealants that are more sensitive to seal parameters i.e. heat seal temperature, pressure and dwell time (Nase et al., 2014), a cohesive peelable sealant is completely welded to the substrate that prevents the chance of leakage, thus the shelf life of the product inside the package is not affected (Liebmann et al., 2012). Cohesive peelable sealants are mainly achieved through blending two or more immiscible polyolefins, such as blends of polyethylene (PE) or ionomer with Polybutene-1 (PB-1) and blends of poly(ethyl methyl acrylate) (PEMA) with propylene-ethylene copolymers (Liebmann et al., 2012). Light weight, low cost, excellent processability and low heat seal initiation temperature are among the key factors which make polyolefins and in particular PE as the best candidates for seal

applications (Coles et al., 2003). However, when used as seal materials, these blends mainly suffer from narrow peelable heat seal temperature window (ΔT_p). A wider ΔT_p facilitates process over a broader temperature range and preserves the peelability even after high temperature processes such as sterilization of the package. Increasing the heat seal temperature enhances the seal strength through enhancing the diffusion of longer polymer chains across the interface of a seal area thus, formation of more perfect crystalline structures upon cooling (Stehling and Meka, 1994; Mueller et al., 1998). Consequently, the seal strength increases up to a point where the peel functionality is lost and the sealant becomes a lock seal. Blending PE based matrices with PB-1 is known to result in peelable sealant with quite broad peelable heat seal temperature range (Nase et al., 2008b) but inconsistent peel performance (Nase et al., 2008a) due to the long term aging of PB-1 after melt process (Kalay and Kalay, 2002, 2003; Yamashita and Kato, 2007; Hu and Tashiro, 2016; Qiao et al., 2016).

Despite the significant breakthroughs of clay polymer nanocomposites (CPN) in a variety of applications (Vaia et al., 1999; Pavlidou and Paspaspyrides, 2008; Zou et al., 2008), there is still high potential to benefit from nanoclays in many emerging applications. Beside light

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weight, low cost and easy-processability, CPN with only a small amount of nanoclay possess very unique properties mainly due to high aspect ratio of nanoclay (Sinha Ray and Okamoto, 2003; Kamel, 2007; Pavlidou and Papaspyrides, 2008; Shokoohi and Arefazar, 2009; Mihindukulasuriya and Lim, 2014). Packaging is an important global industry that has recently exploited nanotechnology to develop innovative products with novel characteristics which are absent from pure polymers or composites (Pereira de Abreu et al., 2007; de Azeredo, 2009; Arora and Padua, 2010; Mihindukulasuriya and Lim, 2014). Montmorillonite (Mt) is the most common type of layered nanoclays (de Azeredo, 2009) and is composed of highly charged silicate layers with very high surface to volume ratio. To reduce the high surface energy arising from high aspect ratio, silicate layers form platen clusters (Arora and Padua, 2010). It is known that a good dispersion of nanoclays is critical to take advantage of superior properties of nanoclays in CPN. However, the poor interactions between hydrophilic clay particles and hydrophobic polymers hinder the diffusion of polymer chains into clay interlayer spaces and its effective dispersion. Numerous studies have investigated the effect of nanoclay surface chemistry on the affinity between the polymer and clay surface and its impact on properties such as thermal (Krump et al., 2006; Lei et al., 2006; Leszczyńska et al., 2007; Sharma and Nayak, 2009; Fukushima et al., 2013; Molinaro et al., 2013; Yourdkhani et al., 2013; Daitx et al., 2015; Shabaniyan et al., 2015), mechanical (Qi et al., 2006; Lee et al., 2007; Rhim et al., 2009; Fukushima et al., 2013; Yourdkhani et al., 2013; Shah et al., 2016), barrier (Kim et al., 2005; Rhim et al., 2009; Molinaro et al., 2013; Yourdkhani et al., 2013), and rheological (Lei et al., 2006; Lee et al., 2007; Jahromi and Khodaii, 2009) of the CPN. Surface modification of nanoclay is usually adequate to improve dispersion of nanoclay in polar polymers such as nylon (Fernes et al., 2004; Okada and Usuki, 2006), but in the case of non-polar polymers such as PE and polypropylene (PP) incorporation of an appropriate compatibilizer that is miscible with the polymer matrix and whose polar groups are adequate to interact with clay surface, is necessary to achieve intercalated clay polymer nanocomposites (Kato et al., 1997; Kawasumi et al., 1997; Okada and Usuki, 2006; Sharma and Nayak, 2009). The performance of CPN are usually enhanced by increasing the dispersion and distribution of nanoclays through compatibilizer addition (Hasegawa et al., 1998; Jacquelot et al., 2006; Zhu et al., 2011, 2014).

A considerable number of CPN studies suggest important benefits of nanoclays in achieving high performance materials. To date, controlling the dispersion and distribution of clay particles in polymer matrix have been shown to enhance various properties of CPN such as thermal, electrical, mechanical, barrier and rheological properties. Nevertheless, it is not clear how the microstructure of nanoclay affects the seal and peel performance of CPN sealants. In a recent study, it was shown that changing the phase localization of nanoclay from the dispersed phase to the interface of PE/polyolefin blends converts the lock seal behavior to peelable behavior (Mohammadi et al., 2018). Manias et al. (2009) and Zhang et al. (2009) reported obtaining peelable clay/PE/ethylene vinyl acetate (EVA) nanocomposite with a versatile peel performance with a heat seal temperature range of about 38 °C. They attributed the observed peelable performance to a synergistic effect of the EVA copolymer and the nanoclay particles. Although their results showed the potential of nanoclay in inducing peelability in PE sealants, it is not clear how the nanoclay microstructure can influence the peel performance of clay/PE nanocomposite films. It should also be noted that they used a very complex system composed of linear low-density PE (LLDPE), low density PE (LDPE), and EVA in the presence of nanoclay which makes it difficult to segregate the effect of nanoclay from the effect of other components.

This work systematically reports on the effect of dispersion and distribution of nanoclays on peel performance of clay/PE (as the most common seal material) nanocomposite sealants. For this aim, the impact of nanoclays surface modification as well as the effect of a PE-g-MA compatibilizer on the dispersion and distribution of nanoclays in the

seal layer and its consequences on the seal and peel properties of the sealants composed of clay/PE nanocomposites were discussed. Finally, a mechanism of peeling correlating with the clay/PE nanocomposite morphology was proposed.

2. Experimental

2.1. Materials

Low density polyethylene (LDPE, Novapol LF-0219-A) was obtained from Nova Chemicals and was used as the sealant layer in this study. Linear low-density polyethylene (LLDPE, Exxon LL3003.32) and high density polyethylene (HDPE, SCLAIR 19A) were obtained from ExxonMobil and Nova Chemicals, respectively, and were used as support layers to eliminate yielding of the seal layer upon peeling. Unmodified sodium Mt (Cloisite Na⁺), organomodified clays Cloisite20 and Cloisite15 were provided by Byk company. The two organomodified nanoclays have a similar organomodifier of dimethyl dihydrogenated tallow in which tallow is approximately 65% C18, 30% C16, 5% C14 with cation exchange capacity (CEC) of 95 and 125 for Cloisite20 and Cloisite15, respectively. A commercial grade of maleic anhydride (MAH) grafted low density polyethylene (PE-g-MA) with a trade name of Bynel 4288 containing 0.23 wt% of MAH was purchased from Dupont and was used as compatibilizer.

2.2. Clay/PE nanocomposite preparation

Highly concentrated (30 wt%) masterbatches of nanoclay/LDPE were prepared using a co-rotating twin-screw extruder (TSE), Leistritz ZSE 18HP, with an L/D ratio of 40. A screw speed of 110 rpm and a temperature profile of 150/160/170/180/180/190/190/190 °C from hopper to die were used for processing. The extrudates were quenched in a cold-water bath, pelletized and dried prior to the next step. In the next step, masterbatches were diluted with LDPE through the same TSE and the same processing conditions to obtain clay/PE nanocomposite containing 6 wt% of different types of nanoclays. For Cloisite15, other clay contents (2, 4 and 10 wt%) were also evaluated. Masterbatches containing the compatibilizer, with compatibilizer/nanoclay ratios of 5:1 and 2:1, were prepared and then diluted with LDPE to obtain samples containing 3 and 6 wt% nanoclay using the TSE at the same processing conditions. PE/Na⁺-6% and PE/C20-6% were used as nomenclatures for clay/PE nanocomposites containing 6 wt% of Cloisite Na⁺ and Cloisite20 respectively and PE/C15-2% to 10% were used as nomenclatures of clay/PE nanocomposites containing 2 to 10 wt% of Cloisite15. PE/PE-g-MA/C15-6% and PE/PE-g-MA/C15-3% are representative of the compatibilized clay/PE nanocomposites with PE-g-MA/nanoclay weight ratio of 2 containing 6 and 3 wt% of Cloisite 15 respectively and PE/PE-g-MA/C15-3% (5:1) is the nomenclature of compatibilized clay/PE nanocomposite containing 3 wt% of Cloisite15 with PE-g-MA/nanoclay weight ratio of 5.

2.3. Film casting

Three-layer films were co-extruded through cast line with single screw extruders, LE20-30 from Labtech, with 5 layer ABCDA 12 in. coat hanger cast film die with a die opening of about 500 μm on average. The films were composed of clay/LDPE nanocomposites as the sealant layer, LLDPE in the middle and HDPE as the outer support layer. The processing conditions of each single screw extruder were as follow: the clay/LDPE nanocomposite sealant was extruded with a screw speed of 40 rpm and a temperature profile of 180/190/195/200, LLDPE and HDPE layers were extruded at screw speeds of 20 and 60 rpm, respectively, and a temperature profile of 180/190/200/210. The polymer films were stretched in air and, then, cooled and collected on calendar rolls chilled with cold water. The collection speed and the draw ratio were manipulated to achieve a uniform tri-layer film with a total

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