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Formulating of a novel polyolefin hazy film and the origins of haze thereof

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Abstract A novel and thin polyolefin hazy film with high haze (>75%), low gloss, and high light transmittance was prepared by the blending polyethylene (PE) with polypropylene (PP) rather than the reverse applying measurements which improved clarity, although Chen and Lue et al. (Annual Technical Conference Proceedings of SPE (2004), pp 2117–2121 and 2140–2143) employed blending to reduce haze. This study also presents the low melt flow rate, high density PE1 blending with PP1, and the blending ratio of 50/50 (wt/wt), are the key factors to prepare the novel polyolefin hazy film. The major origin of total haze of the hazy film was explored. It is found that surface haze is the overwhelming majority of total haze, which is opposite to earlier works about the source of total haze of the films prepared from polymer blend. That rough surface textures was the intrinsic source yielding surface haze, and also huge surface roughness degree (Ra*)* was the reason of the excellent haze properties in the hazy film, was confirmed based on haze measurement, surface topology images and *Ra* data from haze instrument, optical microscopy, and atomic force microscope, respectively.

Keywords Formulating · Polyolefin hazy film · Haze · Surface roughness · Blending · Crystalline

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Introduction

Hazy films are very popular in the package application of high-rate food and books nowadays, and also in the shield employed for advanced large-screen TV and computer display, because of their high haze and paper-like appearance which give faint spectacle reflection and hence little irritation against eyes. This new fashion calls for polyolefin films with excellent haze properties. However, scientists made efforts in eliminating the haze features of polyolefin films [1–9] because the haze appearance of polyolefin films was not desirable in the past. Consequently, researches on promoting haze were relatively little to our knowledge. So the investigation to develop hazy films with the aim of meeting the demand in industry and also interest in scientists' community was conducted.

Usually, there are two routes to obtain or increase haze: one is by internal scatter (called bulk haze usually, also inside, inner or internal haze), and the other by surface scatter (called surface haze usually, also outside haze) because haze is the cloudy or turbid appearance of an otherwise transparent specimen caused by light scattered from within the specimen or from its surfaces. Bulk haze originates mainly from the ununiformity of material inside, such as refraction indexes difference and immiscibility among various polymers [1, 4, 9–22]. The more inhomogeneous the materials are in their bulk, the larger haze they have. Immiscible polymer blend, polymer alloy with refraction index difference, and polymer-filler or polymer-yielding haze agent compound commonly have large internal haze. Researchers recognize universally that internal haze is the main contribution of total haze in the three above materials, and that surface haze is the major source of total haze in the film from individual crystalline polymer. Surface haze only depends on surface roughness which could be achieved by promoting the size and amount of crystalline within polymer film, and second processing, such as chemical etching, embossing, abrading, and adjusting process conditions and so on [9, 21].

Some disadvantages are encountered in using second processing, putting fillers, or adding yielding haze agent into polymer for manufacturing hazy films. Second processing increase post-process, time and cost charge, and are also harmful to mechanical strength of films, while fillers or yielding haze agent depress mechanical properties and light transmittance, even damage completely the light transmittance, of hazy films [24]. So the intrinsic haze property of polyolefin was utilized to avoid the disadvantages. Huck, and many other researchers in this area, reported the films on the base of simple crystalline polymer (individual polymer, namely non-compounding or blending system; in this article, they are called the individual polymer film) and the films manufactured from polymer blend (in this paper they are called the polymer blend film) had inherent haze properties. They also pointed out the total haze of the individual polymer film derived mostly from the surface haze caused by rough surface, while the major source of total haze of the polymer blend film was internal haze [1, 2, 4–6, 10, 12–15, 17–20, 22, 25–36].

If an individual polymer film, namely surface haze caused by rough surface, can meet the haze threshold of commercial hazy films, the formulation and preparing of a new hazy film will be relative simple and ease as compared with selecting a suitable polymer blend system for hazy film. Earlier researches have concluded the surface roughness of a simple crystalline polymer film originated from the formation of crystalline aggregating on or close to the surface of the films and polymer rheological instability generated at the exit of the die, and both were affected by the resin parameters and processing conditions. As a result, it seems that there are both high crystalline polyolefin resin and the suitable processing conditions, in addition with taking reversely Huck et al.'s conclusions and measurements for clarity to enhance haze, the polyolefin hazy film based on individual polyolefin resin will be accomplished at once.

Nevertheless, all the individual PE or PP films, even if adopting adversely Huck et al.'s method to improve haze properties, fail to achieve the haze of 75%, which is a essential requirement of commercial polyolefin hazy film (3-μm thickness). An individual polyolefin film with 3-μm thickness is nearly totally transparent (see Table 5). Since, the haze percent of plastics films decreases with the decrease of the thickness of films [21], we could not produce thin rough films based on simple crystalline polymer, for hazy films. So we could not utilize simply the conclusion of Huck, White, and Wilkes et al. to accomplish a "super-thin" hazy film for meeting market demand.

The results of Tse and Maruhashi et al. [7, 18, 19, 37] demonstrate that polymer blend films have good haze properties, even if the surface of the films is smooth. Bulk haze, hence blending, is introduced to intensify the haze of the individual polyolefin film and break through the restraining of the film thickness in accomplishing ultra-thin hazy films. Polyolefin blend was tentatively applied to prepare a desirable hazy film to combine internal haze with surface haze to promote total haze, although Chen and Lue [23, 38] blent high density PE or low density PE with metallocene linear low density PE (mLLDPE) to reduce haze of the resulting film.

In this article, the design and preparation of polyolefin hazy film is reported because of the scarce literatures of polyolefin hazy films, in the mean while the effects of both molecular weight and crystallizability of PE on haze properties of the polymer blend film is presented and utilized to optimize the formulation of polyolefin hazy films. At last, an unexpected origin of haze which conflicts with previous works [18, 21, 37], existed in the novel polyolefin hazy film, is demonstrated and confirmed.

Materials and methods

Materials

Several polythenes and one polypropylene are used in this work. The melt flow rates (MFI) and density (ρ) of PE resins are provided in Table 1. Polypropylene (PP1) is supported by BP, British, MFI of 5.0 g/10 min, and random copolymer. Silicone oil used in measuring the bulky haze of films, refractive index, $n = 1.512$, is from Kelong Chemical Co., China.

Table 1 Specification of polyethylene resins

PE Resource	PE1 Korea Petrochem. HDPE/Ho-po	PE2 QiLu Petrochem. HDPE/Copo-R	5000S QiLu Petrochem. HDPE/Ho-po	2908 FuShun Petrochem HDPE/Ho-po	1I2A Yanshan Petrochem LDPE	1F7B QiLu Petrochem. LDPE
MFI (g/10 min)	0.1	0.1	1.0	9.0	2.0	7.0
ρ (g/cm$^{3)}$	0.957	0.950	0.964	0.960	0.918	0.920

Ho-po homopolymer, *Copo-R* random copolymer, *HDPE* high density polyethylene, *LDPE* low density polyethylene

Compounding and re-pelletizing

The blends for hazy films were prepared as following: first, a variety of polythene were mixed, at the special blending ratio, with polypropylene (PP1) by hand, and then was extruded by SHJ-20 twin screw extruder (screw diameter of 21 mm, the ratio of length over diameter (L/D) = 32, from Nanjing Giant machinery Co. Ltd., Nanjing) with the extrusion temperature of 170–200 °C and rotation speed of screw of 200 rpm, then pelletized. Control samples made from corresponding individual resins, PEs or PP1, were extruded and re-pelletized following the same conditions.

Blown films processing

The resins re-pelletized were blown into film at Hakke RHEOMEX254 single screw extruder with blown film processing unit (Hakke MessTechnikGmbHu.Co., German), in the range of temperatures from 170 to 200 °C, frost line height of 2.0 cm, blown-up ratio of 3.4, and the thickness of blown films is 26 μm (the thickness of the films in Table 5 is 3 μm).

Optical microscopy (OM)

Optical microscopy (XP-201, Zhoushan Optical instrument Co., Shanghai) was used to take photos of surface topography of the blown films, and the amplification ratio is 250×.

Atomic force microscopy (AFM)

The surface morphology (namely surface texture) of the blown films was observed with an Atomic force microscopy (AFM) (SEIKO Instruments Inc., SPM3800/SPA400, Japan) in tapping mode, and the measuring conditions referred to reference [39]. The surface roughness of the blown films (Ra) was obtained by statistical analysis software supported by SEIKO Instruments Inc. In essence, the Ra represents the mean asperity height of the protrusion on the surface.

Haze measurement

The haze (H) of the blown film samples was tested according to ASTM D1003 by Haze-Gard Plus haze meter (BYK Gardner, German). The haze of blown films immersed into the silicone oil was measured and the haze value was defined as bulky haze (H_b) and correspondingly the haze of films which did not be immersed as total haze (H_t). The surface haze (H_s) equals to the difference between H_t and H_b [1, 22, 25]. Besides haze, the light transmittance (T) also could be recorded in the haze device.

Gloss measurement

The gloss (G) o f the blown film samples was obtained by a Gloss-Gard Plus gloss meter (BYK Gardner, German) in terms of ASTM D2457.

Results and discussions

The formulating of polyolefin hazy film

The feasibility of blending polyolefin to promote haze properties

Employing bulk haze of polymer blend film to promote the haze properties of individual polymer film was proposed in the “Introduction” section, so the validity of this idea was examined first. The data in Tables 2 and 3 are the haze percent of films made from imple polyolefin and polyolefin blend, respectively. It is concluded that the haze percent of the latter is larger than that of the former (blown films produced from corresponding individual polyolefin); for instance, the haze percent

Table 2 Haze properties of the individual polymer films (thickness 26 μm)

	PE1	PE2	5000S	2908	1I2A	1F7B	PP1
H_t (%)	90.3	90.1	23.2	15.3	5.72	4.51	23.7
H_b (%)	23.5	22	9.89	7.65	1.83	1.51	5.1
H_s (%)	66.8	68.1	13.31	7.65	3.89	3.0	18.6
MFI (g/10 min)	0.1	0.1	1.0	9.0	2.0	7.0	5.0

Table 3 Haze properties of the blend polymer films (thickness 26 μm)

	PE1/PP1	PE2/PP1	5000S/PP1	2908/PP1	1I2A/PP1	1F7B/PP1
H_t (%)	99.4	95.2	44.6	42.5	51.2	39.6
H_b (%)	44.1	46	6.5	7.7	10.3	9.3
H_s (%)	55.3	49.2	38.1	34.8	40.9	30.6

The blend ratio of PE/PP1 is 50/50 (wt/wt) of the PE1/PP1 blend film was 99.4, larger than 90.3 of the PE1 film, and 23.7 of the PP1 film as well. The larger the haze of an individual PE film, the better haze properties its corresponding blend film has. In a word, blending resulted in the improvement (increasing) of the haze feature of polyolefin films.

Selecting polythene

According to Huck [1], Stehing [12], Wilkes [22, 32] et al., resin parameters including molecular mass (generally by MFI) and crystallization (usually by the structures of molecular chain) have great influence on the haze of simple polymer films. From Tables 2 and 3, it is found that MFI and crystallizability of resin are also the important factors in yielding haze of the blend films. Increasing MFI of PE, LDPE or HDPE would reduce the total haze of the blend films. The decrease of haze percent is remarkable in blend films and simple resin films when the MFI of PE rise from 0.1 to 9 g/10 min. This is consistent with the results derived from simple crystalline film [4]. The blend film with maximum haze percent is the PE1/PP1 film, which derives from such composition that the PE resin possesses MFI of 0.1 g/10 min, while in the work of Ashizawa et al. [4], the film with maximum haze percent based on the PE resin possessing *MFI* of 1 g/min.

The crystallizability of resin is another important role in yielding haze in individual films, at the same time it shows its power in the blend films. Branched chain, or side group, on PE molecular chain will decrease crystallizability of PE as well as thus haze of the blend films. For LDPE-1I2A and LDPE-1F7B, their molecules contain lots of branched chains with different length, and this would potently suppress the crystallization of PE resin and then haze properties. Thus their corresponding blend films have lower haze percent in our experiment. PE1 and PE2 both possess similar MFI, but PE2 is a copolymer and hence lower crystallization by comparison to PE1, also resulting in lower haze in PE2/PP1 blend film compared with PE1/PP1 blend film.

Finally, the haziest binary film in the case, PE1/PP1 blend film, was selected as the pilot formula of the polyolefin hazy films.

Determining the optimal blending ratio of PE versus PP

The blending ratio of PE/PP selected above, 50/50 (wt), might not be the highly desirable value, and thus further strict experiments is required for determining the optimum blending ratio. The experimental results are presented in Table 4. It was

Table 4 The influence of the ratio of PE1 versus PP1 on the haze of blend films (thickness 26 μm)

	10/90	30/70	40/60	50/50	60/40	80/20
H_t (%)	80.1	98.5	98.7	99.4	98.3	96.4
H_b (%)	31	55.4	51.2	44.1	49	47.1
H_s (%)	49.1	43.1	47.5	55.3	49.3	49.3

The blend ratio of PE1/PP1 is wt/wt

found in the Table 4 that the haze was not sensitive to the blending ratio of PE/PP. The films with blending ratio of 30/70, 40/60, 50/50, and 60/40 (PE/PP, wt/wt) have almost identical haze percent. Only when PE content is very large or small, such as 80 or 10 wt%, the blend film has comparatively small haze percent. However, it was found that for the films with blending ratio of 40/60, 30/70, or 60/40, the haze percent remarkably decreased when their thickness decreased to 3 μm (the reason will be discussed later). Therefore, 50/50 is chosen as the optimum blending ratio of PE1/PP1 for the hazy film.

In the end, the resulting polyolefin hazy film with the thickness of 3 μm, and the corresponding individual resin films in the similar conditions severed as reference samples were produced, and their optical properties (shown in Table 5) were estimated. Table 5 gives also clearly a confirmation of the feasibility of the above formulating. Though the thickness of film is 3 μm, the novel hazy film still possesses haze percent higher than 75.

The origins of haze properties of the polyolefin hazy film

In “The feasibility of blending polyolefin to promote haze properties” section, a remarked increase in haze was confirmed, which poses naturally a question about what is the reasons to justify the increase. In the view of existing general conclusion, the origin of the novel hazy film should also be bulk haze. However from Table 3, it was found that bulky haze in the novel hazy films was lower than the surface haze even if accruing in bulky haze was fairly remarkable due to employing blend. It seems that bulk haze is not a determining source of total haze in the novel hazy film. The haze difference between H_s and H_b in the film was 11.2%, a not significant difference value. In contrast, the difference between H_s and H_b in the individual films was great, such as haze difference of 43.3% in the PE1 film. These facts naturally lead us to suppose the origin of the hazy films may be synergic contribution of bulk haze and surface haze, just being responsible to our original intention combining bulky haze of polymer alloy with surface haze of simple crystalline polymer film to enhance total haze. But data in Table 5 demonstrate clearly that surface haze is much larger, above six times in practical, than bulk haze. That is to say, the surface haze absolutely predominates in the total haze of the hazy film. This conflicts, at least in the apparent, with the conclusion of Tse et al. [19] and Maruhashi et al.’s [37] in which inner haze is the primary contribution in polymer blend films and our common sense concerning this subject.

A spontaneous question was arisen whether the novel hazy film had an unexpected or different origin of haze. So the surface textures were further

Table 5 Optical properties of the hazy film (thickness, 3 μm) and two corresponding simple films

	H_t (%)	H_b (%)	H_s (%)	G (%, hazy surface)	G (%, clear surface)	T (%)
Hazy film	80.5	13.0	67.5	11.3	58.2	90.5
PE1 film	3.25	1.43	1.82	/	92.0	92.2
PP1 film	1.86	1.21	0.65	/	94.3	92.6

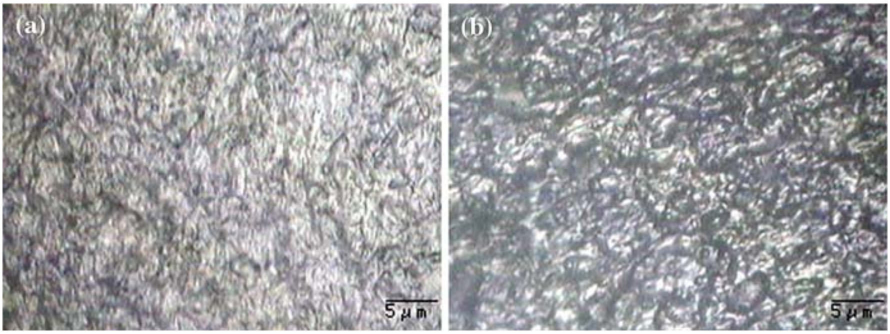

Fig. 1 The surfaces topology of PE1 film and PE1/PP1 (50/50, wt/wt) blend film by optical microscopy, ×250. (**a**) PE1 film, (**b**) PE1/PP1 blend film

examined because surface haze primarily derives from surface scattering, while surface scattering originates from rough surface textures [5, 8, 17, 20, 22, 27, 28, 31–33, 35, 40]. So, surface texture is another reliable means to blow away the above cloud. If surface texture of the hazy film is rougher than that of corresponding individual film and positive relationship between Ra and surface haze is constructed, it could conclude that the determining source of haze in the hazy film is surface haze, at the same time, the hazier one could be picked out by comparing the Ra of the blend films with that of the simple polymer films.

Surface textures could be measured by lots of special methods and meters, whereas optical microscope (OM for short) and atomic force microscopy (AFM for short) are two of instruments used widely nowadays [5, 10, 29, 32, 33, 39]. Before utilizing AFM, the surface topology of the films, PE1 and PE1/PP1 binary blend (Fig. 1) were first obtained by an optical microscope because of its wider visual field which could give us more information and hence higher credit. Obviously, the simple resin film has the smaller and more compact mountain-like topology and lower height of asperity comparing with the blend film, despite blending will damage crystallizability and lessen crystalline agglomerate size, and thus lower surface roughness in theory. The fact that the former is smoother when we touched them may verify further the conclusion. These results lead to that the major parts of total haze of the blend films might be surface haze, like the individual films.

The images of OM cannot give us quantitative, namely precise, the amount of surface roughness of each film owing to the only superficial observation with naked-eye, so picking out the haziest film was conduct in view of AFM.

Figure 2 confirms clearly the deduction concerning the haze origin of the hazy film drawn from Fig. 1. The vertical axis of the tilted image of AFM represents the height of asperity on the surface of film. The height of peaks on the surface of the PE1/PP1 blend film is far beyond those on the surface of PE1 film. It is 1×10^4 nm in the PE1/PP1 blend film whereas 1×10^3 nm in the PE1 film. Besides, it is seen also that the asperity on the PE1/PP1 film is larger than those on the PE1 film. The statistical analysis software of AFM gives the exact value of surface roughness (Ra). The Ra of the surface of PE1/PP1 blend film is 0.59 (Table 6), nearly twice of the surface roughness value of PE1 film (0.34). So the surface scattering of the blend

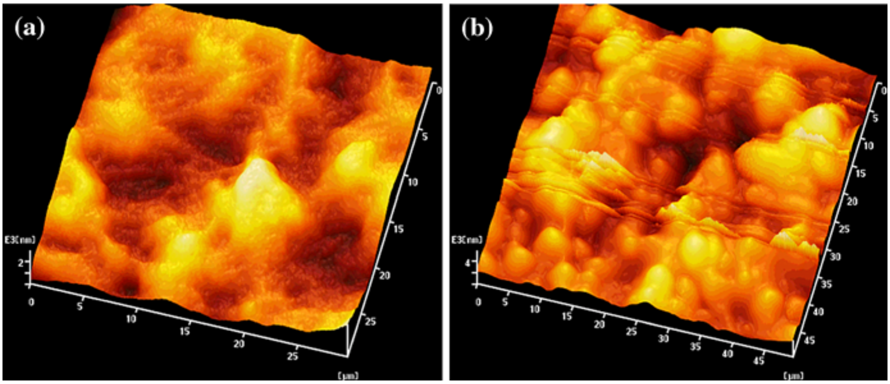

Fig. 2 The surfaces topology of **a** PE1 and **b** PE1/PP1 (50/50, wt/wt) blend film by AFM (scan size, PE1 30 × 30 μm, PE1/PP1 50 × 50 μm)

Table 6 The surface roughness of films calculated from the AFM (thickness 26 μm)

	PE1	PE1/PP1 (50/50, wt/wt)	PE2/PP1 (50/50, wt/wt)
Ra (μm)	0.34	0.59	0.50

films is much larger than that of the corresponding simple resin films, and this fact incorporating with the conclusion derived from Table 5, justifies that the major, even determining, source of total haze of our hazy film is surface haze.

Paul et al. have pointed out the thickness of a film would play an important role in haze, especially in bulky haze. The thinner a film, the smaller haze percent it has [21]. So it is seen the total haze falls down to 80.5% when the thickness decreases to 3 μm (Table 5), compared with 99.4% when thickness is 26 μm (Table 3). At the same time, the bulky haze dramatically decreases to 13.0%, and surface haze increase to 67.5% (from 55.3%). The difference between bulky and surface haze is also absolutely sharp. The thickness of 26 μm is too thick and causes more extra bulky scatter, thus larger inner haze and smaller difference between bulky and surface haze (Table 3).

For further verifying the above conclusion which the major origin of our hazy film is surface haze, the surface textures of other blend films with OM were inspected extensively and explored whether the haziest film selected in the experiment via surface haze was agreed with which by surface roughness, and also that the decrease or increase of surface haze and surface roughness was under the same trend. The optical microscope images of the surface textures of 5000S/PP1, 2908/PP1 and 1I2A/PP1 blend films were presented in Fig. 3 (PE1/PP1 blend film is used as a reference, and other films are not presented for brevity). In "Selecting polythene" section, it is known that with increasing MFI of PE (PE, 0.1 g/10 min; 5000 s, 1 g/10 min; 2908, 9 g/10 min), the surface hazes steep decrease. Virtually, the surface topology of blend films in the experiment correspondingly smooth abruptly with increasing MFI of PE. The PE1/PP1 film has rough, packed granule

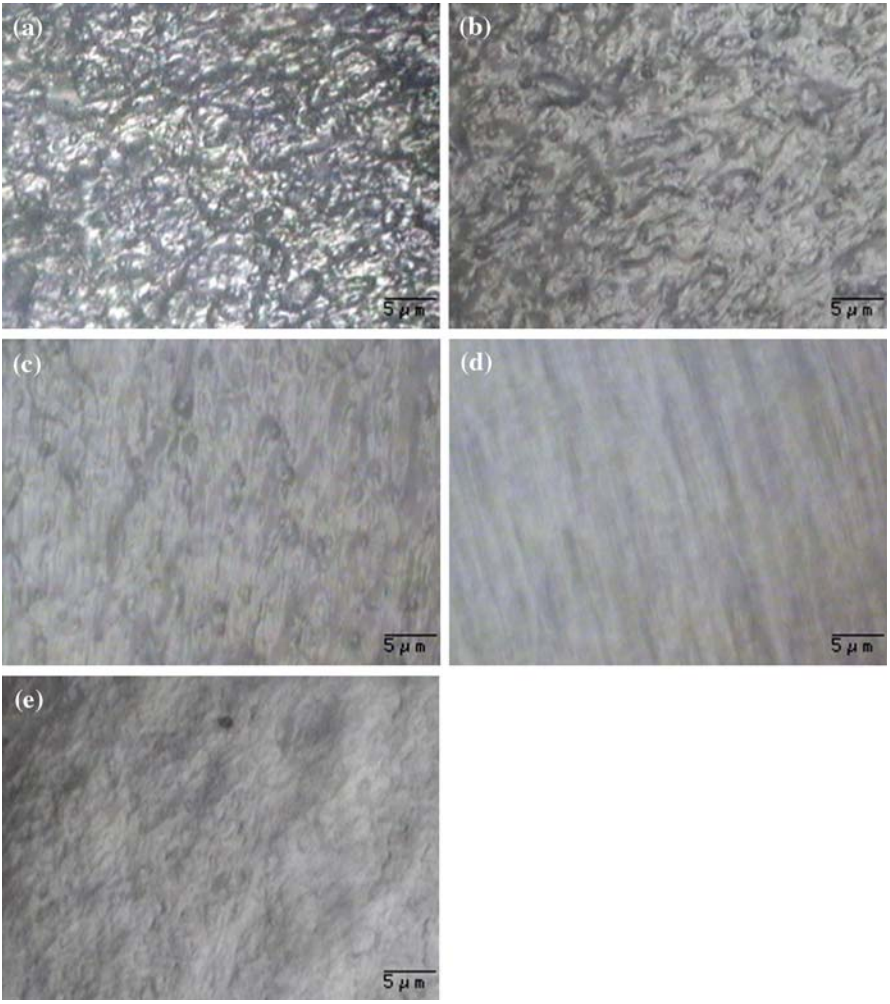

Fig. 3 The surface textures of films different PE blends with PP1 (50/50, wt/wt, ×250, OM). **a** PE1/PP1, **b** PE2/PP1, **c** 5000S/PP1, **d** 2908/PP1, and **e** 1I2A/PP1

surface, while the surface of 5000S/PP1 film is apparently flat and the bump on the surface is barely visible, the 2908/PP1 film nearly totally plain and flat without any asperity except some micro-fibril textures because of flow orientation. At the same time, the 1I2A/PP1 film also holds almost smooth surface and the surface asperity is nearly negligible due to the low crystallization of 1I2A. In a word, when the surface haze decreases or increases, the surface roughness becomes small or large corresponding in our experiment, the surface topology of polymer films agrees well with the haze percent in Table 3, namely, the conclusion which surface haze determines the blend films' total haze is reliable.

It is noted that haze percent in the blend films is not sensitive to blending ratio in "Determining the optimal blending ratio of PE versus PP" section and this may lead

Table 7 Effects of composite ratio on films surface patterns (thickness 26 μm)

	30/70	50/50	60/40
Ra (μm)	0.44	0.59	0.50

The blend ratio of PE1/PP1 is wt/wt

to selecting unsuitable blending ratio. But surface roughness vary keenly with blending ratio, so the surface textures of some blend films with various blending ratio were observed and their surface roughness were measured (Table 7). The surface roughness of the film with blending ratio 60/40 or 30/70 is evidently smaller than that of the films with blending ratio 50/50.

Conclusions

The novel, super-thin polyolefin hazy film with outstanding haze properties and light transmittance was prepared via blending two common polyolefin due to simple employing reversely measurements,which improved clarity (reducing haze) of polymer films failure in enhancing haze. Its haze percent is higher than 75, even if its thickness is down to 3 μm, which is much larger than the individual resin films with hazy appearances. HDPE with large molecular weight and high crystallization is beneficial to increase haze property when it is blent with PP1. That HDPE with MFI of 0.1 g/10 min blend with PP1, blending ratio of 50/50 (wt), was confirmed as preconditions for preparing polyolefin hazy film. That the origin of haze of the novel hazy film was surface haze was confirmed by haze test, surface topology, and surface roughness in view of haze measurement device, OM and AFM, respectively.

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