

고헤이즈(High Haze) 특성을 가진 이축연신 폴리프로필렌/탄산칼슘 필름의 제조 및 성능 연구

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Preparation of a Biaxially Oriented Polypropylene (BOPP)/CaCO₃ Film with High Haze and Its Performance Study

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Abstract: To explore the feasibility and potential advantages of utilizing inexpensive and readily available calcium carbonate (CaCO₃) in the preparation of biaxially oriented polypropylene (BOPP) matting films, a polypropylene (PP)/CaCO₃ composite cast film was prepared and then stretched using a biaxial tensile testing machine. The impact of CaCO₃ content on crystallinity, matting, and mechanical properties of the composite films was investigated. When CaCO₃ was added at 35 wt%, the haze reached as high as 89.6%, showing excellent matting properties. Moreover, it was observed that either excessive or insufficient CaCO₃ leads to a reduction in crystallinity. The surface morphology of the composite films was investigated using a scanning electron microscope (SEM). The film's roughness was improved by the CaCO₃ dispersion on the surface. This study provides an effective reference for the advancement of the plastic film industry in regard to superior packaging materials.

Keywords: biaxially oriented polypropylene, calcium carbonate, matting film, haze.

Introduction

Biaxially oriented polypropylene (BOPP) films have been widely used in packaging, labels, and other applications owing to their superior physical stability, gas barrier properties, clarity, gloss, and resistance to abrasion.¹⁻⁴ The optical properties such as haze are essential directions for their development. The matting film has considerable attention in the packaging field due to its high haze, soft reflected light, and good printing effect.^{5,6}

When illuminated by incident light, the light undergoes multiple refractions as it traverses the crystalline and amorphous region domains of the film, accompanied by scattering events. These processes reduce the intensity of the incident light and

augment scattering, thereby contributing to light loss.⁷ The microstructure of the film surface is a significant factor affecting the extinction performance of the film. When incident light hits a rough surface, specular reflection is replaced by diffuse reflection. Diffuse reflection results in the interference of reflected light with each other, thereby enhancing the intensity of the scattered field. This weakens the intensity of specular reflection, achieving the goal of extinction.^{8,9} The primary approach to enhance the surface micromorphology is by utilizing blending modification to either cast and compound the matting agent onto the film surface, or by adhering the prepared matting agent to the film surface via surface coating.^{10,11} Moreover, the coating method suffers from several limitations, such as complex processing, high cost, and potential delamination of the coating.¹²⁻¹⁴ In contrast, the preparation of matting films by blending and casting modification is simple and effective, and eliminates the risk of coating delamination. This simplifies the production process and shortens

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the cycle time, making it highly suitable for industrial scale-up. Currently, the materials used as matting agents can be categorized into two categories, namely organic matting agents and inorganic matting agents. Most commonly, organic matting agents are synthetic wax and polymer microspheres.¹⁵⁻¹⁸ Inorganic matting agents include mainly synthetic silica, titanium dioxide, diatomite, etc.¹⁹⁻²²

Calcium carbonate (CaCO₃) is an abundant mineral, accounting for 5% of the earth's crust. It is used as a functional filler and coating pigment for paper, rubber, plastic, and adhesive.²³⁻²⁵ Doping of CaCO₃ usually improves the mechanical, thermal, and barrier properties of composites.²⁶⁻²⁸ Therefore, CaCO₃ is also used to manufacture film products such as polyurethane films, seaweed biopolymer films, and isotactic polypropylene (PP) composite films.²⁹⁻³² The dispersion of CaCO₃ increases the surface roughness of the film, which results in an extinction effect. Liu *et al.*³³ prepared flexible nanocellulose-based composite films which exhibited high hydrophobicity and a haze degree of up to 84.8% through the amidation of carboxylated transparent oxide nanocellulose with octadecyl amine and the *in-situ* growth of amorphous CaCO₃. These films were utilized for high-efficiency light management in solar cells. Arkadiusz Kloziński *et al.*¹¹ investigated the effects of modified and unmodified CaCO₃ additions on the PP extrusion processes and the selected functional properties of cast composite films. They observed that increasing the filler content led to a proportional increase in film haze.

In order to further expand the application of CaCO₃ in the matting film, we compounded PP/CaCO₃ matting masterbatch onto the PP surface using a multi-layer cast extruder. The matting film was then obtained by bidirectional stretching with a high-temperature bidirectional tensile tester. Furthermore, the effects of CaCO₃ content on the properties of matting, surface morphology, mechanical properties, and crystallization behavior of BOPP composite films were investigated.

Experimental

The Preparation of Masterbatch. A commercial PP HP550J with a melt flow rate of 3.11 g/10 min (2.16 kg at 230 °C) was used as a polymer matrix supplied by Zhejiang Petrochemical Co., Limited. PP and CaCO₃ particles with an average particle size of 1.5 μm were placed in a blast drying oven at 80 °C for 12 h. Then, a 30% mass fraction of PP resin was added to a 70% mass fraction of CaCO₃, and the resulting mixture was blended in an extruder. Extrusion and feeding speeds were 150 rpm and

Table 1. PP/CaCO₃ Composite Films Composed of Different CaCO₃ Particle Contents

Sample	CaCO ₃ (wt%)	PP (wt%)
PP	0%	100%
PP-7%CaCO ₃	7%	93%
PP-14%CaCO ₃	14%	86%
PP-21%CaCO ₃	21%	79%
PP-28%CaCO ₃	28%	72%
PP-35%CaCO ₃	35%	65%
PP-42%CaCO ₃	42%	58%

8 rpm, respectively. The five temperature ranges of the extruder were set at 170, 225, 235, 235, and 235 °C. After extrusion, cutting, drying, and mixing the material evenly, we finally obtained the PP/CaCO₃ matting masterbatch.

The Preparation of Cast Film. The matting masterbatch was cast using a five-layer co-extrusion casting machine (BP8178B-V). The surface layer of the film consisted of a blend of PP and CaCO₃ particles, while the base layer was composed of PP. The ingredient ratio is shown in Table 1. During extrusion, the matting masterbatch was in contact with the surface air, and the PP was in contact with the cooling roller (Figure 1). The extrusion temperatures of all five screw extruders were 170/225/235/235/235 °C, and the die head was 235 °C. The speed of the twin-screw extruder A was 10 rpm. The speeds of the single-screw extruders B/C/D and E were 150 and 100 rpm, respectively. The matting layer accounts for 15%. After being cooled by the cooling roller, the sheet was rolled up by the traction roller to form a thick sheet. Finally, a PP/CaCO₃ composite cast film was obtained.

The Preparation of BOPP/CaCO₃ Films. The PP/CaCO₃ composite cast film was made by casting and then cutting into a 9 cm × 9 cm sample. It was placed into a biaxial tensile testing machine (BSE770) for simultaneous stretching, and was stretched simultaneously both in the machine direction (MD) and transverse direction (TD) directions with a stretching ratio of 5×7 (MD×TD). The stretching preheating time was 180 s, the stretching temperature was 170 °C, the stretching rate was 10 mm/s, and the heat setting time was 10 s. Finally, BOPP/CaCO₃ films were obtained.

Differential Scanning Calorimetry (DSC). To analyze the crystallization and melting temperature (*T_m*) of polymer materials, the differential scanning calorimetry (DSC) measurements were carried out using a DSC1/500, Switzerland. The mass of the film sample was 5 mg. Under nitrogen atmo-

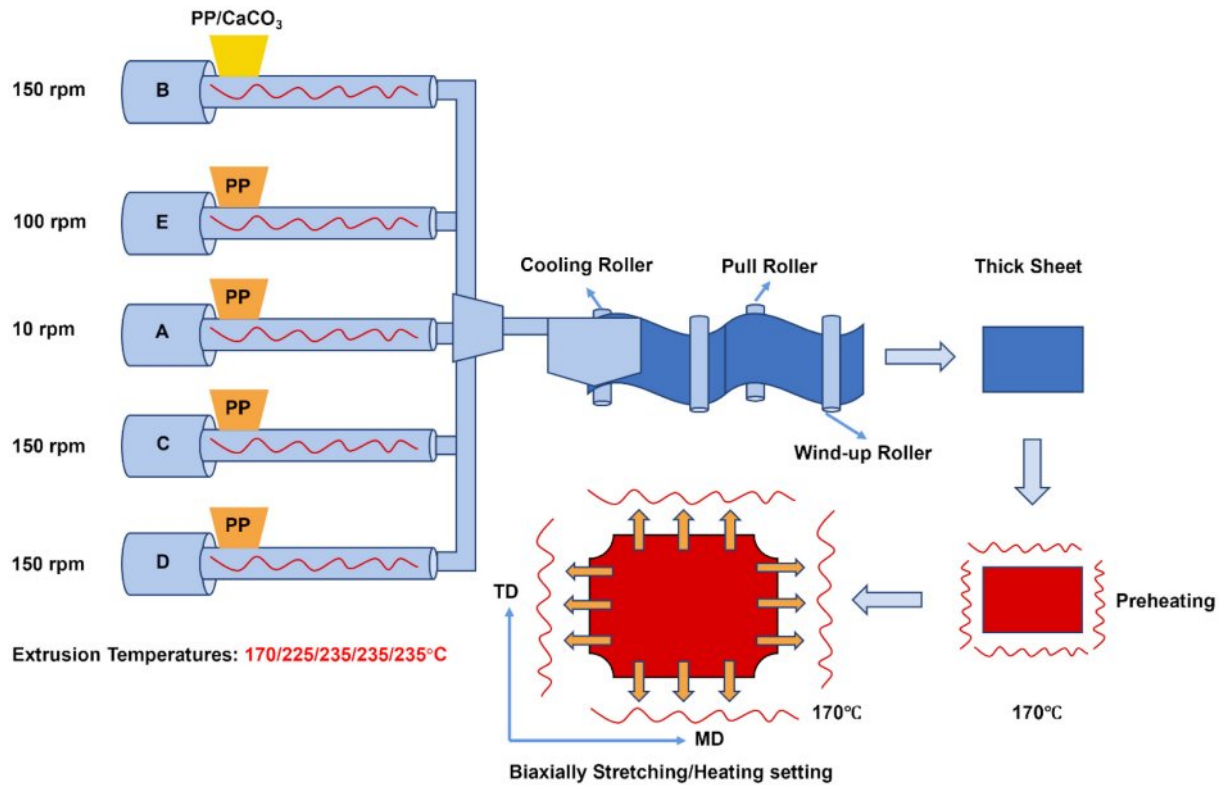


Figure 1. The preparation process of BOPP/CaCO₃ films.

sphere, it was heated from 20 to 200 °C at a heating rate of 20 °C/min, and the DSC heating curve was recorded. The crystallinity (x_c) of the film was calculated by Formula (1):

$$x_c = \frac{\Delta H_m}{w_f \Delta H_m^0} \times 100\% \quad (1)$$

w_f is the mass fraction of film PP, and ΔH_m is the melting enthalpy of PP in the sample. ΔH_m^0 is the melting enthalpy of 100% crystallized PP, and the value is 209 J/g.

Morphological Analysis. The surface morphology of the film sample was observed with a scanning electron microscope (SEM, ZEISS GeminiSEM 300). Before SEM observations, the samples were made conductive by gold sputtering.

Haze and Light Transmittance Analysis. The film was cut into 100 mm × 100 mm sized samples and tested using a TH-09 transmittance/haze tester.

Mechanical Property Analysis. The film was cut along the MD direction into a long strip with a 200 mm × 20 mm width, and the initial distance between the clamps was 100 mm ± 2 mm. An ETM-D universal tensile testing machine was employed to evaluate the mechanical properties of the film specimens at a test speed of 100 mm/min. Each film sample was tested 5 times, and the average value was taken.

Results and Discussion

Crystallization Properties of BOPP/CaCO₃ Composite Film. The DSC heating curve of the BOPP/CaCO₃ composite matting film, and the corresponding thermodynamic parameters are shown in Figure 2, and Table 2, respectively. The addition of CaCO₃ particles gradually enhanced the crystallinity and melting temperature of polypropylene, and improved the thermal stability of the composite film within the range of 7 to 35 wt%. The maximum values of crystallinity and melting temperature of the BOPP/CaCO₃ composite film at 35 wt% were 45.0% and 166.1 °C, respectively. These values were 5.7% and 1.2 °C higher than those at 7 wt%. However, when the CaCO₃ addition amount is 42 wt%, the crystallinity of the BOPP/CaCO₃ composite film decreases to 39.1%. This is due to the fact that the agglomeration of CaCO₃ leads to a decrease in nucleation, resulting in a specific reduction in crystallinity.

The addition of CaCO₃ particles results in the melting temperatures of the BOPP/CaCO₃ composite film ranging from 164.4 to 166.1 °C, indicating the α crystal form of BOPP. This demonstrates that the addition of CaCO₃ particles to BOPP does not alter the crystal structure of composite films. The melting temperature of the BOPP film without adding CaCO₃ particles

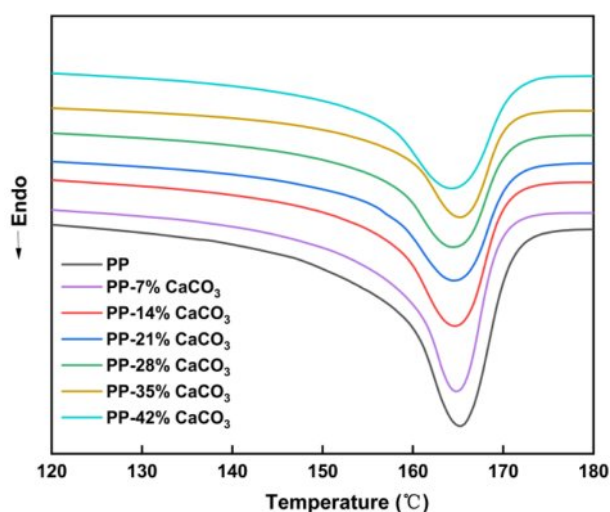


Figure 2. DSC curve of BOPP/CaCO₃ composite matting film.

Table 2. Crystallization Parameters of BOPP/CaCO₃ Film

Sample	T_m (°C)	ΔH_m (J/g)	x_c (%)
PP	165.8	97.0	46.4
PP-7%CaCO ₃	164.9	81.5	39.3
PP-14%CaCO ₃	165.2	81.2	39.6
PP-21%CaCO ₃	165.6	84.7	41.8
PP-28%CaCO ₃	165.7	86.1	43.1
PP-35%CaCO ₃	166.1	89.3	45.0
PP-42%CaCO ₃	164.4	76.5	39.1

is 165.8 °C. When the amount of CaCO₃ particles added to the BOPP film is 7 wt%, the melting temperature is 164.9 °C, which is 0.9 °C lower than that of the film without CaCO₃ particles. Because the addition of low-content CaCO₃ particles reduces the

crystallization of BOPP molecules, thereby lowering the melting temperature of the film. Moreover, a small amount of CaCO₃ particles can act as a nucleating agent, they provide fewer nucleation sites. The interface area between PP and CaCO₃ particles is also smaller, resulting in a decrease in crystallinity (39.3%).³⁴

Optical Properties of BOPP/CaCO₃ Composite Film. Figure 3 is a line chart showing the haze and transmittance of CaCO₃ particle content on the BOPP/CaCO₃ composite film. The BOPP film without CaCO₃ particles possesses higher light transmittance, weak matting performance, and low haze. As the amount of CaCO₃ particles added to the surface film increases, the haze of the BOPP/CaCO₃ composite film displays an upward trend, and the light transmittance begins to decrease. When the additional amount is 35%, the haze of the BOPP/CaCO₃ composite film is 89.6%, indicating an exceptional matting effect. Although this value is lower than the 96.8% haze reported by Arkadiusz *et al.*¹¹ for an unstretched 120 μm thick film, it still remains at a sufficiently high level for commercial applications. When the amount of CaCO₃ added to the surface layer reaches 42%, the haze decreases to 88.5%. This phenomenon can be attributed to the agglomeration of CaCO₃ resulting from excessive CaCO₃ addition, resulting in an uneven haze on the film surface. It reveals that the addition of CaCO₃ can markedly improve the matting performance of PP/CaCO₃ composite film.

Micromorphology Analysis of BOPP/CaCO₃ Composite Film. Figure 4 shows the SEM images of BOPP/CaCO₃ composite films with different contents of BOPP/CaCO₃ particles. The surface of BOPP film without CaCO₃ is smooth. After the addition of CaCO₃, the surface film's smoothness gradually

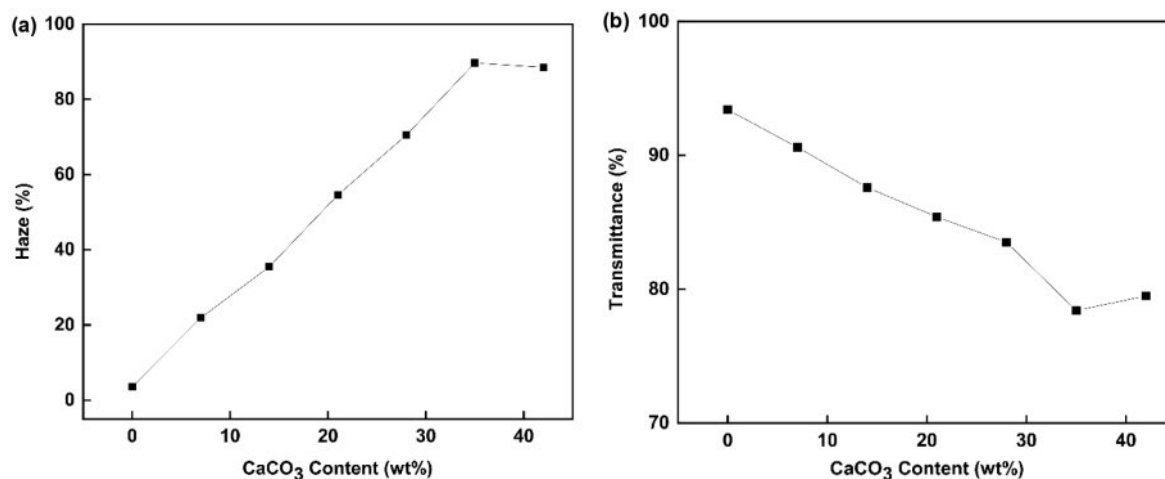


Figure 3. Effects of BOPP/CaCO₃ composite films with different CaCO₃ contents: (a) haze; (b) transmittance.

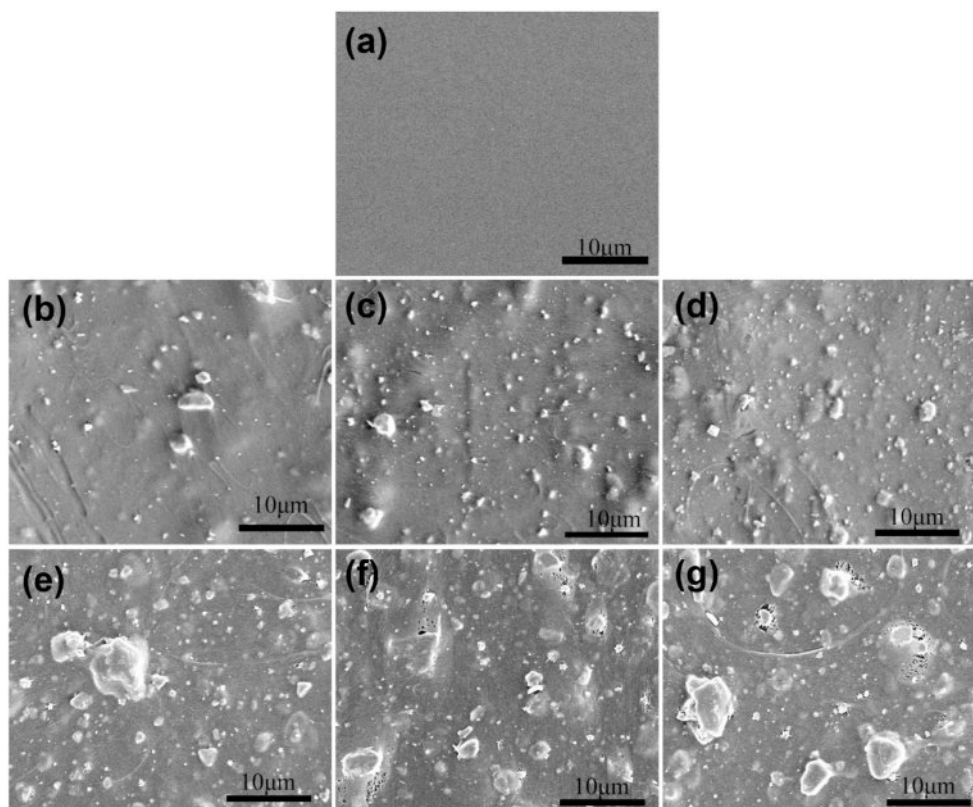


Figure 4. SEM images of BOPP/CaCO₃ composite films with different contents of CaCO₃: (a) 0 wt%; (b) 7 wt%; (c) 14 wt%; (d) 21 wt%; (e) 28 wt%; (f) 35 wt%; (g) 42 wt%.

decreases, its roughness becomes evident, and the inhomogeneity and agglomeration of CaCO₃ particles progressively increase. From the optical properties of the film, it is apparent that as the additional amount of CaCO₃ particles increases, the haze also increases. Therefore, it is beneficial to enhance the extinction degree by improving the roughness of the film surface.

To further investigate the impact of surface roughness on haze, a roughness tester was used to measure the changing trend of surface roughness of BOPP/CaCO₃ composite films (Figure 5). The roughness tends to increase with the addition of CaCO₃ particles, which is consistent with the change in film haze in Figure 3(a). When the content of CaCO₃ particles is 35 wt%, the film roughness reaches a maximum value of 0.53 μm. Similarly, when CaCO₃ particles were added at 42 wt%, the film's roughness and haze decreased due to the large amount of CaCO₃ agglomeration. Therefore, an appropriate amount of CaCO₃ particles uniformly distributed on the surface of the film can improve the film's roughness and haze, as well as its matting properties. The hydroxyl groups on the CaCO₃ surface can be reacted with coupling agents, unsaturated organic acids, and surfactants to enhance dispersibility and compatibility.^{35,36}

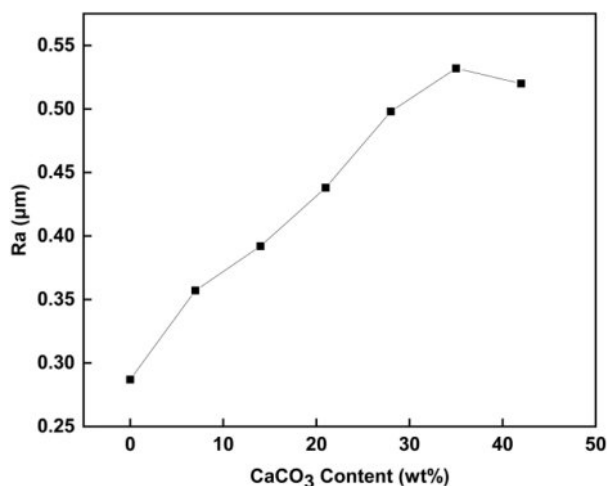


Figure 5. Effect of CaCO₃ content on the roughness of BOPP composite films.

Mechanical Properties of BOPP/CaCO₃ Composite Film.

It can be seen from Figure 6 that the addition of CaCO₃ to the PP matrix leads to a decrease in the tensile strength of BOPP/CaCO₃ composite films. The tensile strength of BOPP/CaCO₃ films in the TD direction decreases from 51.4 to 42.3 MPa

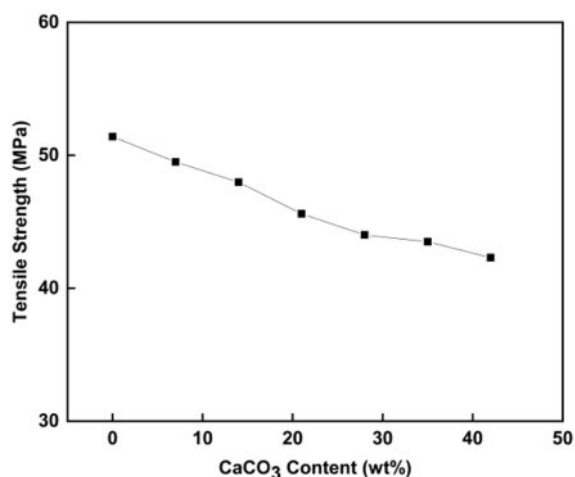


Figure 6. Effect of CaCO₃ content on the tensile strength of BOPP composite films.

upon an increase in surface CaCO₃ content from 0 to 42 wt%. Nevertheless, this value still surpasses the tensile strength of 15 MPa reported by Arkadiusz *et al.*¹¹ for films with 30% CaCO₃, and exceeds the maximum tensile strength of 28.38 MPa documented in Praditham's work.³⁷ The superior mechanical performance observed in our study can be attributed to the ultra-thin film thickness (approximately 18 μm) achieved *via* biaxial stretching, which enhances the polypropylene film's mechanical properties through molecular orientation. The diminution in tensile strength may be attributable to an inadequate interfacial adhesion between CaCO₃ particles and the PP matrix, as well as insufficient stress transfer at the interface.^{38,39}

Figure 7 illustrates that the elongation at the break of the BOPP/CaCO₃ composite film decreases with the increase of CaCO₃

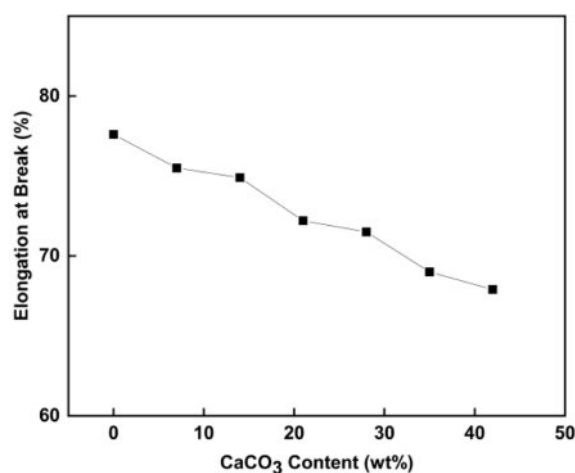


Figure 7. Effect of CaCO₃ content on elongation at break of BOPP composite films.

particle content. At a CaCO₃ particle addition of 35 wt%, the elongation at break was 69%, which is still within the acceptable range. This could be explained by a large number of CaCO₃ particles forming phase separation from the PP matrix, causing holes in the surface film and reducing the elongation at break and toughness of the BOPP/CaCO₃ composite film. As can be observed in the SEM images (Figure 4), higher CaCO₃ content promotes particle agglomeration. These agglomerates act as stress concentration points within the matrix, facilitating crack initiation and propagation, as also reported in previous studies.^{40,41} During the biaxial stretching process, these CaCO₃ agglomerates are prone to detach from the PP matrix, resulting in the formation of micro-voids. These defects ultimately reduce the elongation at break and overall toughness of the composite film.

Conclusions

This study employed a multi-layer cast extruder to fabricate thick multi-layer PP/CaCO₃ composite sheets. Then, a biaxial tensile testing machine was used to simultaneously biaxially stretch the thick sheets to prepare a BOPP/CaCO₃ composite film. The effects of different CaCO₃ additions on optical, micromorphological, mechanical, and thermal properties of BOPP/CaCO₃ composite films were investigated. Upon increasing the concentration of CaCO₃ particles to 35 wt%, agglomeration phenomena occurred on the surface of the film, leading to an augmentation in surface roughness and a subsequent decrease in light transmittance. The film exhibits enhanced diffuse reflection, achieving a high haze value of 89.6%, indicative of a superior matting effect. In this research, we have extended the application of inexpensive and readily available CaCO₃ in BOPP matting film, lending concrete support for the advancement of the plastic film industry and meeting the market demand for superior packaging materials. Finally, long-term stability, such as moisture resistance, weather resistance, and durability, is crucial for packaging applications. The findings of this study suggest that films with a lower and uniformly dispersed CaCO₃ content hold promising potential for enhanced thermal stability and durability. A systematic evaluation of these long-term properties through comprehensive aging studies will be a valuable direction for our future research.

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Conflict of Interest: The authors declare that they have no conflicts of interest.

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