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Influence of Processing Conditions and Composition on Dispersion Behaviour of Nano Calcium Carbonate in Polypropylene Matrix

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Abstract

Compounding of filled thermoplastics is a common method for improving mechanical properties of plastics. In this way, the use of nano fillers makes it accessible to improve mechanical properties. In this paper, first, the mechanical properties of different polypropylene (PP) composites containing micro and nano calcium carbonate were investigated. Then, a well dispersed compound filled with nano calcium carbonate was prepared using a specific formulation by a twin screw extruder. Consequently, various samples including different content of nano fillers were prepared. The dispersion behavior of nano particles in PP matrix was analyzed using a scanning electron microscopy (SEM). In order to verify the effect of nano calcium carbonate on mechanical properties, tensile and impact experiments were carried out. The obtained results revealed that using dispersed nano calcium carbonate in filled PP results in improving mechanical properties.

Keywords: Composite – Polypropylene – Dispersion - Nano Calcium carbonate – Particle size.

Introduction

In recent years, several investigations have been performed on the effect of calcium carbonate content on mechanical properties of nano composites. In the case of mineral fillers there are literatures about the effect of calcium carbonate content on mechanical properties. Chan and his coworkers [1]. have been studied the effect of using various filler content on modulus, tensile strength and impact strength. They indicated that using 4% of nano calcium carbonate in the matrix resulted in increasing modulus. However, increase of nano calcium carbonate content made no significant change in tensile modulus and even higher filler content led to decrease tensile modulus. Furthermore, they reported that impact strength improved by introducing nano calcium carbonate. Baoqing et al. [2] investigated the effect of oleic acid surfaced modified calcium carbonate nano particles on physical and mechanical properties of nano composite. They found that addition of 2% Oleic acid treated nano calcium carbonate resulted in achieving the optimum properties and increasing the impact strength by 60%. While, increase of filler content leads to fall down flexural strength of composite. TEM images illustrated that calcium carbonate grafted oleic acid nano particles dispersed properly in the PP matrix. Yang et al. examined the properties of PP/CC nano composites with different concentration of nano particles, in homo and copolymer polypropylene matrix. They observed an intense increase in impact strength of copolypropylene[3]. Lam et al. worked on surface modified nano calcium carbonate treated by sodium stearate and observed a 30% increase in young modulus[4].

Lin et al. focused on the graft content of stearic acid on nano CC. They revealed that use of 20% nano filler with 5.3% graft content as an optimum composition increase the impact strength of a homo polypropylene (*Mn=55000 g/mol*) up to 25%. Their results showed that this formulation leads to a 40% modulus enhancement. Their results showed that addition of 5% nano particle to copolypropylene (*Mn=87000 g/mol*) increase the impact strength up to 80%. They believe that this phenomenon is related to the critical ligament thickness and represent that critical ligament thickness is highly dependent on physical properties of polymer and specially molecular weight[5], [6].

In the case of rheological behavior Karamipour et al. performed a study on the effect of nano calcium

carbonate particles on rheological and dynamic mechanical properties of polypropylene matrix. Their results showed that storage modulus do not changes significantly by adding nano particles. but phenomenological models deserves at least a 5% increase in storage modulus. This difference is due to the weak interaction between polymer and nano particles. On the other hand, loss modulus growth is more than 40% by adding 5% nano particle. Their results revealed that loss factor decreased by increasing the filler content and this decrease is more significant in lower frequencies. Rheological tests show an increase in complex viscosity (η^*) by adding more nano particles[7].

This study focused on the mechanical properties of polypropylene filled with micro and nano sized calcium carbonate particles.

Experimental

Materials

A commercially available block copolymer polypropylene M1600 (MFI 25g/10min) was supplied by LG petrochemical company and was used as the polymer matrix. An antioxidant Irganox 1010 and a polypropylene grafted maleic anhydride, CA100 produced by Orevac were used. Two calcium carbonate grades supplied by Omya Co. possessing the mean particle size of 5 µm and 70nm, respectively.

Sample preparation

Polymer and filler were physically mixed up and fed into a twin-screw extruder (SHJ-36 Nanjing), L/D=40. The samples formulation have been shown in Table 1. The samples PPnc-2/3/4 were prepared by introducing various quantity of two pre-dispersed masterbatch into polymer matrix. The temperature profile was set from 190^{0}C to 220^{0}C, and also screw speed was regulated 350 rpm. Afterwards, experimental specimens were produced using a laboratory injection molding machine at 200^{0}C.

Experiment

Flexural and impact mechanical tests were performed. Flexural test performed by a universal AL7000M GOTECH mechanical tester according to ASTM D790 and Impact test were performed according to ISO 180 standard. All experiments were performed in 23±2^{0}C.

Table 1 Formulation details of samples

MB20% CCNANO	MB10% CCNANO	Nano CC	Micro CC	Anti-Oxidant	PP-G-MAH	PP	
						100	PP
			4	1		95	PPmc
		4		1		95	PPnc-1
	40			1		59	PPnc-2
	40			1	2	57	PPnc-3
20				1	2	77	PPnc-4

Results and Discussion

Impact Strength

Impact experiment was performed at two temperatures 23 and -20℃. Results show that there is no considerable difference in impact strength of micro composite compared to neat PP, as it is illustrated in Fig.1 and Fig.2. Moreover, the sample containing 4 wt% nano calcium carbonate prepared via direct method shows a little increase in impact strength. However, the impact strength of samples PPnc-2/3/4 in which nano particles was introduced in matrix through pre-dispersed masterbatch has been improved. The increase of impact strength is more substantial in nano composites containing polypropylene grafted maleic anhydride.

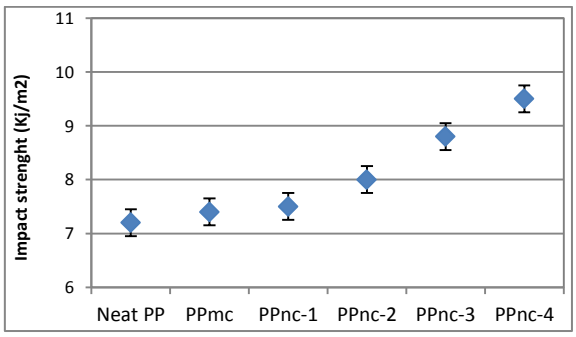

Figure 1 Impact strength of different samples at 23^{0}C

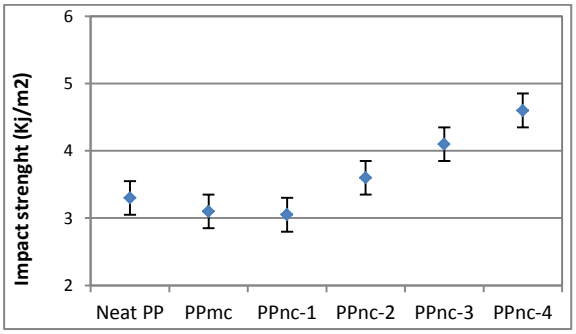

Figure 2 Impact strength of different samples at -20^{0}C

Besides, results revealed similar trend for low temperature impact strength of composites. It can be concluded that the dispersion behavior of nano particles and filler-polymer interaction in samples PPnc-3/4 is more appropriate compared to other composites. This enhancement in impact properties is a consequence of introducing polypropylene grafted maleic anhydride.

Furthermore, impact properties can be confirmed by flexural modulus results, as shown in Fig.3. Flexural modulus has been considerably raised up by introducing a pre-dispersed masterbatch containing 20 wt% nano particles. It seems that adding polypropylene grafted maleic anhydride resulted in better wetting of nano particles by polymer matrix. Consequently, a strong bonding makes between nano particles and polymer chains which lead to better dispersion of nano particle through matrix. Moreover, higher concentration of filler causes to higher tension and consequently better dispersion happens.

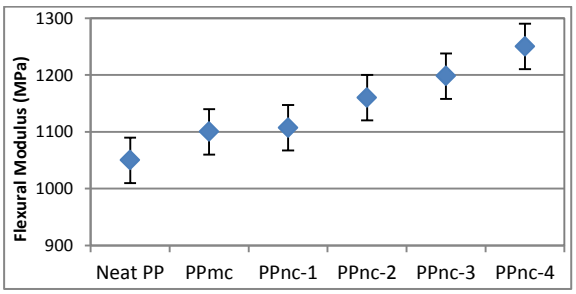

Figure 3 Flexural modulus of different composites

SEM

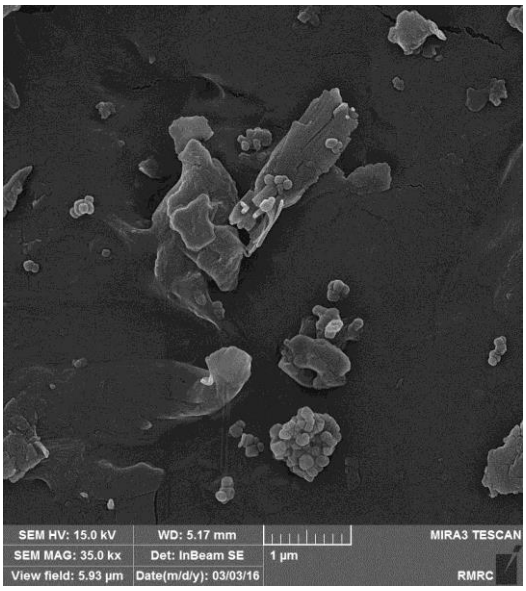

Figure 4 SEM Image of sample PPnc-4

Analysis of sample 4 was conducted to analyze the dispersion quality of nano calcium carbonate in PP matrix. Fig.4 reveals that an appropriate dispersion of nano particles in PP matrix has happened. Moreover, it is clear that a little formed agglomeration exists in matrix, but the size of agglomeration particles is in nano scale.

Conclusion

Use of different formulation and diverse processing methods figured out that utilizing polypropylene grafted maleic anhydride results in wetting and dispersion nano calcium carbonate through polypropylene matrix, and also improves the filler-polymer interaction. Furthermore, it was observed that better dispersion behavior achieved by introducing pre-dispersed Masterbatch. Therefore, use of a masterbatch containing 20 wt% calcium carbonate provides better mechanical and impact properties due to higher tension.

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