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Mechanical and thermal properties of Homo-PP/GF/$CaCO_3$ hybrid nanocomposites

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Abstract. In an attempt to reach a balance of performances in homo-polypropylene based system, the effects of single and hybrid reinforcements inclusions comprising calcium carbonate nanoparticles (2, 4 and 6 phc) and glass fibers (10 wt.%) on the mechanical and thermal properties were investigated. Different samples were prepared by employing twin-screw extruder and injection molding machine. In morphological studies, the uniform distribution of glass fibers in PP matrix, relative adhesion between glass fibers and polymer, and existence of nanoparticles in polymer matrix were observed. PP/$CaCO_3$ (6 phc) as compared to pure PP and PP/GF had superior tensile and flexural strengths, impact resistance and deformation temperature under load (DTUL). PP/GF/$CaCO_3$ (6 phc) composite displayed comparable tensile and flexural strengths and impact resistance to neat PP, while its tensile and flexural moduli and deformation temperature under load (DTUL) were 436%, 99% and 26℃ greater respectively. The maximum impact resistance was observed in PP/CaCO3(6 phc). The highest DTUL was perceived in PP hybrid nanocomposite containing 10 wt.% glass fiber and 4 phc $CaCO_3$ nanoparticle.

Keywords: hybrid composites; homo-polypropylene; glass fiber; $CaCO_3$ nanoparticles; mechanical and thermal behavior

1. Introduction

Polypropylene (PP) benefits from ease of processing, low cost and good resistances to solvents, stress cracking and moisture absorption (Bryce 1997, Hudgin 2006). However, when polypropylene is compared to engineering polymers, it has weaker mechanical and thermal properties. Many efforts have been made to enhance PP performances. Incorporations of 20 wt.%, 25 vol.% and 30 wt.% of short glass fibers into polypropylene led to the 188%, 400% and 321% increases of elastic modulus and 156%, 56% and 185% rises of tensile strength respectively (Yoshida *et al*. 2006, Fu *et al*. 2000, Hufenbach *et al*. 2012). The main reason of aforementioned improvements in tensile properties was attributed to the proper adhesion between glass fibers and polypropylene (Yoshida *et al*. 2006, Fu *et al*. 2000). Nonetheless, the existence of glass fibers in

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polymer matrix may cause significant anisotropic shrinkage and residual stress in molded parts along with severe wear of production equipment (Bryce 1997). Some researchers have investigated the effects of micron-scale mineral particles on the mechanical properties of polypropylene. Zebarjad *et al.* (2004), indicated that adding 30 wt.% of calcium carbonate micron-particles to polypropylene led to 19% decrease in tensile strength, 94% drop in elongation at break and 77% increase in elastic modulus. They argued that the differences in deformations of calcium carbonate particles and polypropylene and their poor interfacial adhesion could lead to easy and quick debonding of calcium carbonate particles from polypropylene during tensile test. Leong *et al.* (2004), observed that the impact strength of polypropylene containing 30 wt.% of talc particles has about 11% reduction as compared with pure polypropylene. They believed this reduction in impact strength could be due to the immobilization of molecular chains of PP because of the existence of plate-like talc particles. Jahani (2011) characterized the mechanical properties of polypropylene/mica and polypropylene/talc composites. The notched-impact strength was reduced about 54 and 26% by applying 50 wt.% of talc and mica mineral fillers respectively. Moreover, about 12 and 10% reduction in tensile strength and 320 and 290% increments in elastic modulus observed by applying 50 wt.% of talc and mica mineral fillers respectively. The reduction of impact and tensile strengths were ascribed to the sheet-form structures of talc and mica particles and hence their stress concentration effects.

Nanocomposites have attracted tremendous interests over the past decades. A large number of researches have shown that mineral nano-fillers could improve mechanical properties of polymers (Sun *et al.* 2009). Adding calcium carbonate nanoparticles to polypropylene may lead to impact strength and tensile modulus improvements (Sun *et al.* 2009, Afshar *et al.* 2010). However, nanoparticles are prone to agglomeration because of their high specific area and surface energy which this may lead to the reduction of tensile and impact strengths. This necessitate the incorporation of surface modifier (Zhang *et al.* 2004, Zokaei *et al.* 2007). Phuong *et al.* (2008) observed 15% reduction in impact strength of PP/6 wt.% clay nanocomposite as compared to pure PP. The decrease in impact strength was attributed to the agglomeration of clay nanoparticles and subsequently the decrease of interfacial contact between nanoparticles and polymer matrix. Lai *et al.* (2009) indicated that adding 6 phr of clay nano-particle to polypropylene in the presence of PP-g-MAH compatibilizer caused 46% increase in elastic modulus, 43% rise in tensile strength and 78% reduction in the elongation at break as compared to pure polypropylene. They believed the presence of compatibilizer had substantial effect on the strength of the interphase layer formed between PP and nano-clay which this may lead to the tensile strength improvement. Bhuiyan *et al.* (2013) studied the tensile properties of PP/CNT nanocomposites. Increases of 22% in the tensile strength and 36% in the elastic modulus were observed in PP/CNT (5 wt.%) nanocomposite. The agglomeration and random orientation of CNT in PP matrix prevented from obtaining better tensile performances as those expected. Moreover, a number of researchers have studied the mechanical performances of composites containing two-scale reinforcements. Arao *et al.* (2012) investigated the mechanical properties of PP/CF (20wt.%)/CNT (1wt.%) hybrid nanocomposite containing 4wt.% of PP-g-MAH compatibilizer. They observed about 15% increase in elastic modulus, 23% increase in the tensile strength and 11% increase in the flexural strength in hybrid nano-composite as compared to PP/ CF (20 wt.%) composite. They argued that the high modulus of CNT, the fairly appropriate adhesion between the matrix and CNT due to the existence of compatibilizer, and the improvement of crystallization of polymer matrix because of the CNT nucleating effect are the main reasons in improving the mechanical properties. Furthermore, they believed the length of CNT and the interaction between CNT and CF are crucial in determining mechanical performances. Saches *et al.* (2013) compared the effects of 1.6 wt.% micron and nano-scales silica particles on the mechanical properties of PP/GF/silica hybrid composite. The results indicated 0.31 GPa increase in elastic modulus and about 10% increment in impact resistance of hybrid nanocomposite as compared to those of microcomposite. The rise of impact resistance in hybrid nanocomposite was attributed to the elevation of absorbed energy in the debonding process of glass fibers from polymer matrix.

This research aimed to investigate the mechanical and thermal performances of homo-polypropylene based system containing single and two-scale reinforcements comprising short glass fibers and $CaCO_3$ nanoparticles. Glass fiber was used at low loading in order to raise the tensile and flexural resistances while avoiding high anisotropic shrinkage and warpage. $CaCO_3$ nanoparticle was employed because of its high specific area, impact modifying effect and ease of dispersion. In this respect, composites composed of homo-PP, glass fibers (10 wt.%) and different concentrations of calcium carbonate nanoparticles (2, 4 and 6 phc) were prepared by melt compounding and injection molding process. The mechanical and thermal properties comprising tensile, flexural and impact resistances and DTUL[1] were assessed. The morphologies were characterized using SEM.

2. Experimental

2.1 Materials

In this research, homo-polypropylene with melt flow index of 25 gr/10 min (230°C and 2.16 kg) produced by Maroon petrochemical company with commercial trademark of Z30S was used as matrix phase. Short glass fibers of 13 microns diameter and 3.2 to 4.8 mm long, surface coated with the coupling agent of Amino Silane (144a ,Taiwan glass group) was employed. The calcium carbonate nanoparticles of 5 to 15 nanometer diameter, surface coated by stearic acid (NPCC-201, Nano Material, Singapore) were incorporated. MAPP with concentration of 0.5-1 wt.% of MAH (Exxelor Po1020, Exxonmobile Chemical, USA) was used as compatibilizer.

2.2 Equipment

A co-rotating twin-screw extruder with the screw diameter of 20 mm and length to diameter ratio of 40 (Brabender, Germany) was used for melt mixing. The standard specimens of tensile, flexural, notched Izod impact and DTUL tests were molded by injection molding machine (Billion H 485/140 TP, France). The tensile and flexural tests were conducted by employing a universal machine (AI-7000M, GOTECH) and the impact tests were implemented by SANTAM-SIT20D impact tester. The deformation temperature under load assessments were performed by employing DTUL tester (XRW-300, Jinjian). Scanning electron microscopy was conducted by using VEGAII TESCAN (Czech Republic).

2.3 Methods

Raw materials comprising polypropylene, glass fibers, nanoparticles and compatibilizer were

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[1] Deformation Temperature Under Load

Table 1 Formulations of different samples

Sample	Components contents			
	PP (wt.%)[1]	GF (wt.%)	PP-g-MAH(phc)[2]	nano-$CaCO_3$ (phc)
Pure PP	100	0	0	0
PP/2C	100	0	1	2
PP/4C	100	0	2	4
PP/6C	100	0	3	6
PP/GF	90	10	2	0
PP/GF/2C	90	10	4.5	2
PP/GF/4C	90	10	6	4
PP/GF/6C	90	10	7.5	6

[1] weight percent
[2] part per hundred compound

dried under vacuum at 80°C for 17 hours. Different compounds were first mechanically mixed and then melt blended by using twin-screw extruder. The speed of extruder's screws was 100 rpm and the temperature of the heaters from feed zone to the die were set on 160, 170, 180, 190 and 200°C, respectively. The abbreviations and the weight ratios of various compounds are indicated in Table1. The standard specimens of tensile, flexural, notched Izod impact and DTUL tests were prepared by injection molding, according to ISO 527, ISO 178, ISO 180 and ASTM D648 respectively, under the melt temperature of 200°C and the mold temperature of 80°C. Prior to molding of standard specimens, the compounds were dried under vacuum at 80°C for 6 hours. Tensile tests were accomplished according to ISO 527 and at the speed of 50 mm/min. The elastic modulus was obtain by using two points of stress-strain diagram corresponding to 0.0005 and 0.0025 mm/mm of strains. The flexural test was accomplished conforming to ISO 178 standard and with supports distance of 48 mm, speed of 2 mm/min and end point of 12 mm. The Izod impact test was conducted according to ISO 180 and under adjusted energy of 11 Joules. All mechanical properties tests were performed at room temperature and each test was repeated 3 times. DTUL tests were carried out according to ASTM D648 and under the stress of 0.445 MPa, heating rate of 120°C/h and end point of 0.25 mm. To observe the extent of matrix-fibers adhesion, to characterize the existence and dispersion of nanoparticles in the polymer matrix, and to study the fracture topographies, SEM images of 63x, 1.2kx and 30kx magnifications were taken from fracture sections of tensile and impact specimens.

3. Results and Discussions

3.1 Morphological characterizations

Fig. 1 indicates the SEM images (63x) taken from the cross sections of tensile test specimens for pure PP and compounds containing various amounts of calcium carbonate nanoparticles. Those compounds containing calcium carbonate nanoparticles (Figs. 1(b) to (d)) when compared to pure polypropylene (Fig. 1(a)) have smoother topography. Fig. 2, shows the SEM images (63x) of compounds containing 10 wt.% glass fibers and different amounts of calcium carbonate nanoparticles (0, 2, 4 and 6 phc). Uniform distributions of glass fibers in PP matrix are observed (Figs. 2 (a) to (d)). Indications of fibers pull-out and the relative adhesion between glass fibers and polymer matrix are perceived in Fig. 3. Fig. 4 depicts the SEM images (30kx) of pure polypropylene and compound containing 4 phc of calcium carbonate nanoparticles. According to Fig. 4(b), the presence of nanoparticles can be observed.

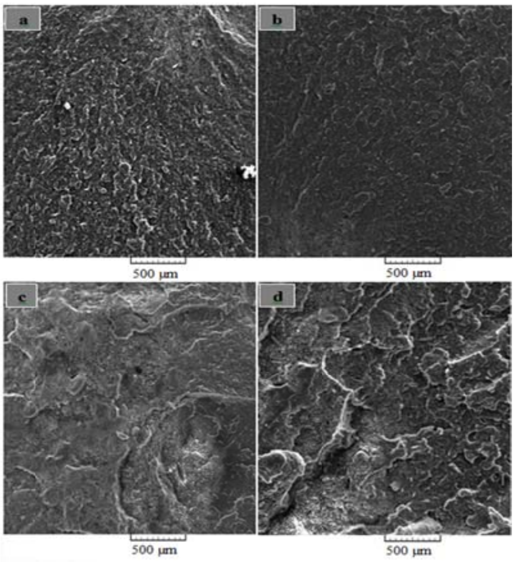

Fig. 1 SEM images of fractured surfaces of tensile test specimens (a) Pure PP, (b) PP/2C, (c) PP/4C and (d) PP/6C

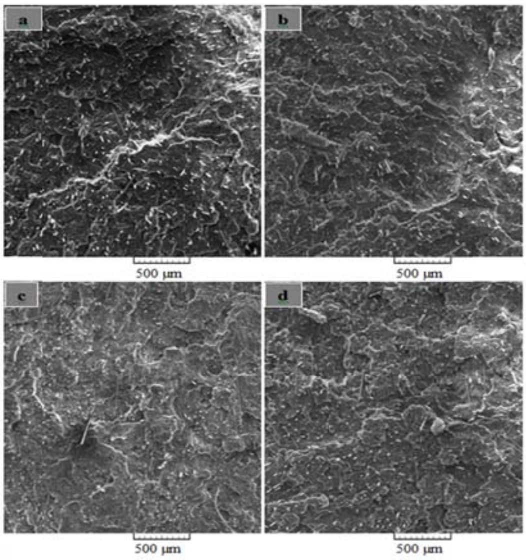

Fig. 2 SEM images of fractured surfaces of tensile test specimens (a) PP/GF, (b) PP/GF/2C, (c) PP/GF/4C and (d) PP/GF/6C

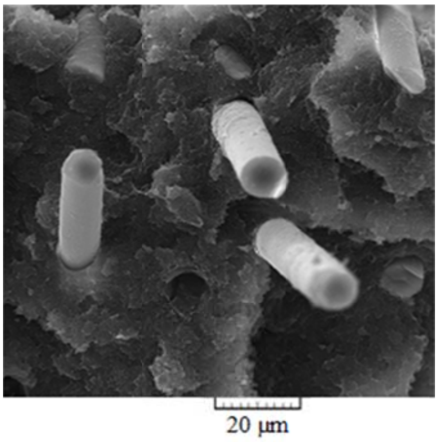

Fig. 3 SEM image of fractured surface for PP/GF tensile specimen

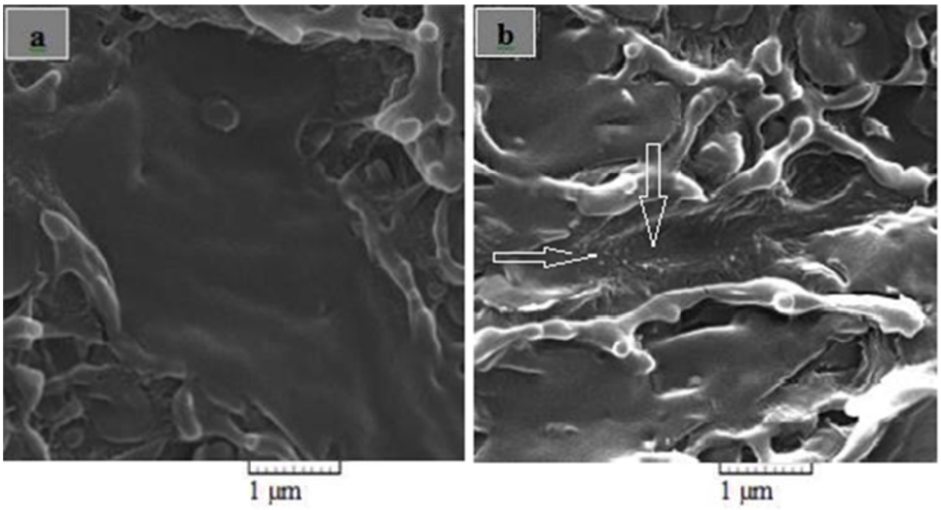

Fig. 4 SEM images of fractured surfaces for impact test specimens (a) Pure PP and (b) PP/4C

3.2 Mechanical properties

Table 2, indicates the results of mechanical assessments comprising tensile, flexural and notch Izod impact tests. Incorporation of $CaCO_3$ nanoparticles and glass fibers led to the substantial increment of modulus. The maximum tensile modulus was achieved in PP/GF/4C composite which was about 447% greater as compared to that of pure polypropylene. The modulus of calcium carbonate nanoparticles is much higher than that of matrix polymer's modulus, hence incorporation of nanoparticles leads to the increase of tensile modulus (Xie *et al.* 2004). Moreover, relatively proper distribution of nanoparticles in polymer matrix and adhesion of polymer-nanoparticle could also increase the tensile modulus (Charifou *et al.* 2015). In addition to abovementioned causes, the increase in crystallinity rate of polymer matrix in the presence of nanoparticles, because of crystal nucleating effect of nanoparticles, could be another reason of tensile modulus growth (Mikesova *et al.* 2014). Incorporation of glass fibers in pure polypropylene induced a large increase in tensile modulus. This can be due to high modulus of glass fibers and appropriate adhesion between fibers and matrix (Hufenbach *et al.* 2012). Fig. 3 shows the relative adhesion between fibers and PP matrix. The depletion of modulus in PP/6C and PP/GF/6C may be attributed to the agglomeration of nanoparticles and application of high amount of compatibilizer. By formation of mineral particles agglomeration, the specific surface area of nanoparticles and their interfacial contact to polymer decrease which may lead to the reduction of tensile modulus (Lin *et al.* 2011). Moreover, the reduction of tensile modulus as a result of adding PP-g-MAH compatibilizer can be attributed to the low modulus of compatibilizer when compared to that of polymer matrix (Ishak *et al.* 2008). According to table 2, inclusion of $CaCO_3$ nanoparticles and glass fibers slightly raised the tensile strength. The maximum tensile strength was obtained in PP/$CaCO_3$ (4 phc). The increase of tensile strength in corresponding nanocomposite could be because of crystal nucleation effect of nanoparticles (Lin *et al.* 2011). The lack of a noticeable increase in tensile strength in compounds containing glass fibers compared to compounds without glass fibers, could be due to the breakage of fibers during melt compounding. By decreasing the fibers length, the interaction between fibers and matrix decreases and consequently a significant increase in tensile strength does not occur (Fu *et al.* 2000, Thomason *et al.* 2002).

The flexural modulus elevated significantly by applying nanoparticles and glass fibers (Table 2). The greatest flexural modulus is achieved in PP/GF/6C which is 99 and 18% superior when compared to those of pure polypropylene and PP/GF respectively. The adhesion between matrix and minerals can limit the polymer chains movement and subsequently raises the rigidity (Agrawal *et al.* 2007). The applications of nanoparticles and glass fibers marginally increased the flexural strength.

According to Table 2, adding 2 and 4 phc of nanoparticles to polypropylene led to the reduction of impact strength. This can be ascribed to the crystal nucleating effect of $CaCO_3$ nanoparticle and subsequently the increment of crystallinity in PP matrix. Crystalline regions absorb less impact energy than that of amorphous regions (Shelesh-Nezhad and Orang 2011). By increasing the nanoparticle loading to 6 phc, the impact resistance elevated as compared with neat PP. Kemal *et al.* reported that the raise of toughness and impact energy may be attributed to enhanced microvoid formations initiated by nanoparticles, which locally deform the matrix surrounding the particles and initiate mass plastic deformation (Kemal *et al.* 2009). The increment of impact strength in PP/$CaCO_3$ (6 phc) may also be attributed to the higher inclusion of PP-g-MAH compatibilizer as compared with those of PP/$CaCO_3$ (2 phc) and PP/$CaCO_3$ (4 phc). The presence of mineral particles could also dissipate impact energy by pinning and bowing mechanisms (Fu *et al.* 2008). Those compounds containing glass fibers exhibited less impact resistance. This result may be due to the formation and growth of cracks in glass fiber tips that act as stress concentrations (Fu *et al.* 2000).

3.3 Deformation temperature under load (DTUL) assessments

Table 2 shows the DTUL tests results for pure polypropylene and various compounds. As nanoparticles content increases, the DTUL increases. In the compound containing 6 phc calcium carbonate nanoparticles, 32% increment in DTUL is observed when compared to that of pure PP. This increment can be related to the crystal nucleating effect of nanoparticles which may lead to crystallinity elevation and consequently increase of rigidity and flexural strength (zhang *et al.* 2004). Moreover, the high modulus of nanoparticle and its low sensitivity to heat as compared to

Table 2 Mechanical properties and deformation temperature under loads of different samples

Sample	Tensile Modulus (MPa)	Tensile Strength (MPa)	Elongation at Break (%)	Flexural Modulus (MPa)	Flexural Strength (MPa)	Impact Strength (KJ/m^2)	DTUL (˚C)
Pure PP	1052.64 ±61.50	22.54 ±0.77	22.29 ±0.72	843.00 ±33.05	14.62 ±0.13	2.85 ±0.07	55.9
PP/2C	3682.56 ±439.90	24.82 ±0.72	9.32 ±0.35	982.00 ±26.55	15.88 ±0.26	2.66 ±0.15	69.8
PP/4C	4625.60 ±279	25.48 ±0.14	8.38 ±0.34	1133.64 ±44.35	16.74 ±0.37	2.25 ±0.28	68.9
PP/6C	4224.80 ±335.70	24.48 ±0.53	7.51 ±0.67	1198.79 ±20.06	16.68 ±0.89	3.05 ±0.14	73.5
PP/GF	4961.28 ±292.60	22.86 ±0.18	7.05 ±0.62	1424.00 ±28.93	14.69 ±0.90	2.19 ±0.18	73.1
PP/GF/2C	4883.76 ±116	22.95 ±0.12	8.17 ±0.17	1453.00 ±29.87	14.95 ±1.06	2.27 ±0.33	67.2
PP/GF/4C	5758.47 ±291	23.25 ±0.11	7.23 ±0.45	1484.00 ±54.34	15.67 ±0.94	1.72 ±0.12	89.6
PP/GF/6C	5642.48 ±522.80	23.66 ±0.34	6.59 ±0.14	1678.00 ±16.10	15.61 ±0.02	2.66 ±0.19	81.9

pure PP, may further improve the deformation temperature under load. In the specimen containing glass fibers, a 31% increase in deformation temperature under load is observed when compared to pure PP. This increment could be attributed to the stress transfer established within matrix-fibers as well as load bearing capability of fibers at higher temperatures (Folkes *et al.* 1982). According to Table 2, for PP/GF/4C compound, the DTUL is increased about 60% and 23% in comparison to those of pure PP and PP/GF respectively. In PP/GF/6C sample, the DTUL was reduced about 9% as compared to that of PP/GF/4C. This is ascribed to the agglomeration of nanoparticles at higher content.

4. Conclusions

The effects of glass fibers and $CaCO_3$ nanoparticles on mechanical and heat performances of homo-PP based composites were experimentally studied. Applications of 10 wt.% glass fibers and 2 to 6 phc of $CaCO_3$ nanoparticles into neat PP noticeably elevated the tensile and flexural moduli. The greatest tensile and flexural moduli were found to be in PP/GF/$CaCO_3$ (4phc) and PP/GF/$CaCO_3$ (6phc) respectively. The maximum tensile and flexural strengths were observed in PP/$CaCO_3$ (4phc). PP/$CaCO_3$ (6phc) nanocomposite when compared to pure PP and PP/GF(10 wt.%) displayed greater tensile and flexural strengths, impact resistance and deformation temperature under load. PP/GF/$CaCO_3$ (6phc) hybrid nanocomposite exhibited comparable tensile and flexural strengths and impact resistance to neat PP, whereas its tensile and flexural moduli and DTUL were 436%, 99% and 26°C larger respectively. The maximum impact resistance was perceived in PP/$CaCO_3$ (6phc). The highest DTUL was observed in PP hybrid nanocomposite containing 10 wt.% glass fiber and 4 phc $CaCO_3$ nanoparticle. Stiffness, dimension, concentration and dispersion conditions of CaCO3 nanoparticles and glass fibers along with their interactions with PP matrix were perceived to be the major factors affecting mechanical and heat properties of PP based composites.

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