

# Investigation of Mechanical, Thermal and Melt Flow Performance of Polycarbonate Hybrid Composites Containing Mica Flakes and Glass Fiber

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Glass fiber (GF) and mica (MC) filled polycarbonate (PC) composites were fabricated using extrusion process due to considerable applications of PC as recyclable and thermo-stable material in mainly medical, packaging and transportation fields. Tensile, hardness and impact tests, differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA), melt flow index (MFI) test and scanning electron microscopy (SEM) findings were reported. Mechanical test results show that tensile strength and modulus are improved for higher GF and lower MC contents. The necking behavior of PC disappear after MC and GF inclusions due to the reduction of ductility. Impact strength of PC is reduced with both additions of GF and MC. Glass transition temperatures ( $T_g$ ) of PC and relevant composites were estimated by the help of DSC analysis.  $T_g$  of PC shifts to higher temperatures by the addition of GF. According to TGA results, GF and MC additions cause remarkable increase in thermal stability of PC. MFI of PC extends up to higher values with increase in GF concentration. SEM analysis reveals that GF and MC display nonhomogeneous dispersion for their high loading ratios. Individual inclusions of additives yield higher mechanical and thermal performance than hybrid composites.

## Introduction

Polycarbonate (PC) is an engineering plastic that has high toughness and transparency. Carbonate segment in its structure yields ductility whereas high heat stability comes from the bisphenol A segment. PC is a thermoplastic polymer which has ability to ease of recyclability. Therefore, it is used in a broad range of industrial areas including glazing, electronics, medical devices, transportation applications, food contact packaging and household appliances [1-3].

Glass fibers (GF) are mainly composed of silica and alumina and they are conventionally used as reinforcing agent in polymer composites due to having high modulus and strength [4-6]. Short glass fiber is considered as favorable additive for thermoplastics because of providing good strength, low weight and recyclability in addition to processability by conventional techniques such as extrusion and injection molding [7,8]. The final properties of short GF filled thermoplastic composites depend on several parameters including concentration, length and aspect ratio of GF as well as adhesion and orientation of fibers to the polymer matrix [9-12]. Various research works regarding GF reinforced PC have been studied in literature which improvements in tensile strength, modulus and hardness, whereas reduction in toughness, impact strength and elongation were reported [13-18].

Mica is the general name for aluminosilicate minerals of the phyllosilicate family. Reinforcement in a plane structure is the advantage of the using mica in polymer composites. Polymers gain excellent properties in terms of rigidity, dielectric property, chemical and fire resistance via introduction of mica [19-22].

Hybrid composites contain at least two different fillers and they are designed to obtain multifunctional materials thanks to synergy between filler phases [23-28]. MC and GF have been chosen as additives in order to achieve synergistic effect on thermal and mechanical improvements of PC-based composite materials. There have been a limited number of research works that deal with polymer composites including MC and GF at the same time [29,30]. According to literature survey, this research is the first study that focus on the fabrication of GF and MC reinforced PC hybrid composites. The main aim of this study is the development of recyclable, mechanically strong and thermally stable PC-based composites due to their wide range of applications as medical devices, transportation and packaging. In this contribution, influence of additions of short GF and MC together to mechanical, thermal and morphological performance of PC composites. Tensile, Shore hardness and impact tests, thermal gravimetric analysis (TGA), melt flow index (MFI) test and scanning electron microscopy

(SEM) results are reported. Findings are discussed according to observations by the means of alignment of individual GF and dispersion of MC flakes into polymer matrix.

## Experimental

### Materials

The commercial PC was supplied from Sabic, UAE under the trade name of Lexan LS2. Linear mold shrinkage and density of PC are 0.5% and 1.2 g cm<sup>-3</sup>. GF was purchased as chopped form and commercially silane sized by Şişecam AŞ, Turkey. The commercial name of GF is PA 1. The length, diameter and ultimate strength values of individual GF are 13 µm, 4.75 mm and 3.45 GPa respectively, supplied by the manufacturer. MC was obtained from Omya Mining, Turkey under the trade name of Mica 900. The mean diameter of MC flakes was reported as 900 µm by supplier.

### Preparation of composites

Before extrusion process, PC, GF, and mica were dried under vacuum at 80°C for 24 hours. The percent compositions of fillers for PC/GF and PC/MC composites were 5%, 10%, 20%, and 30%. Total loading level of hybrid composites were kept constant at 30% by weight due to composites containing additives with this composition yield the best performances in previous studies [29-32]. The percentages of hybrid composites (MC%/GF%) were 5/25, 10/20, 20/10, 25/5. The prepared dry mixtures were processed via a co-rotating twin screw micro-compounder (DSM Xplore, Netherlands) for 8 minutes. The screw speed and process temperature were 100 rpm and 280°C, respectively. The obtained extrudates were cut into small pieces and then dried under vacuum at 80°C before injection moulding. For mechanical tests, the specimens were shaped by a laboratory scale injection-molding device (Micro-injector, Daca Instruments). The barrel and mold temperatures were 290°C and 80°C, respectively. The required pressure for injection was set to 5 bar. Test specimens were obtained from injection molding as the dog-bone shape with dimensions of 7.4×2.0×80 mm<sup>3</sup>.

### Characterization methods

The tensile properties were investigated using Lloyd LR 30 K (West Sussex, UK) universal tensile testing machine. The load cell was 5 kN and crosshead speed was 10 cm min<sup>-1</sup> (ISO 527-2-5a). The tests were conducted on dog-bone shaped specimens. Tensile strength, percent elongation and tensile modulus values were recorded. The results are the average value of five tests. Charpy impact energy values of PC and composites were measured using Ceast Resil Impactor (Torino, Italy) with the pendulum of 4J according to ASTM D256-92 standard. Recorded results represent an average value of at least five samples with standard deviations. Shore D hardness tests were

performed by Zwick digital hardness tester (Ulm, Germany) according to standard of ISO 7619-1. Thermal stability of samples was analyzed by TGA 2950, TA Instruments (New Castle, USA). TGA tests were done in the temperature range of 20°C to 550°C with the heating rate of 10°C min<sup>-1</sup> under nitrogen flow of 50ml min<sup>-1</sup>. Differential scanning calorimetry (DSC) measurements were studied by using a Perkin Elmer (Massachusetts, USA) Diamond DSC at a scanning rate of 10°C min<sup>-1</sup> between 50°C-300°C temperature range under N<sub>2</sub> atmosphere. MFI values were measured using Coesfeld Meltfixer LT (Dortmund, Germany). Test measurements were carried out under the specified load of 2.16 kg at the process temperature of 280°C. Weighted melt flow rate results represent average values of at least ten samples with standard deviations for each composites. Morphological characterizations of composites were examined by JSM-6400 field emission scanning electron microscope (JEOL Ltd, Tokyo, Japan). Surfaces of fractured samples obtained from impact test were coated with a thin layer of gold in order to obtain a conductive surface. SEM micrographs were taken at x250 and x2500 magnifications.

## Results and discussion

### Tensile test

The characteristic stress–strain curves of PC/MC, PC/MC-GF hybrid and PC/GF composites are represented in **Fig. 1** and the relevant tensile test data are listed in **Table 1**. As seen from **Fig. 1** that unfilled PC displays necking behavior after ultimate strength associated with its ductility. GF addition for the lowest concentration (5%) exhibits necking with shorter strains relative to PC. This behavior is not present for other composites due to decrease of ductility after incorporation of MC and GF.

**Table 1.** Tensile test data of PC and composites.

Samples	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Modulus (GPa)
PC	60.7±2.2	14.4±1.3	0.92±0.03
PC/GF 5	62.6±1.7	12.9±1.4	0.97±0.06
PC/GF 10	70.0±2.4	11.1±1.1	1.09±0.04
PC/GF 20	88.4±1.8	10.9±1.1	1.34±0.05
PC/GF 30	76.6±1.6	9.4±1.0	1.28±0.06
PC/MC 5–GF 25	86.3±2.0	8.0±0.8	1.21±0.05
PC/MC 10–GF 20	74.0±1.4	9.3±0.7	1.08±0.03
PC/MC 20–GF 10	62.2±1.6	9.7±0.9	0.95±0.06
PC/MC 25–GF 5	56.3±1.8	10.3±0.6	0.91±0.04
PC/MC 5	88.8±3.7	7.7±1.0	1.56±0.12
PC/MC 10	42.7±2.4	6.3±0.7	0.96±0.08
PC/MC 20	21.8±1.8	8.2±0.8	0.78±0.06
PC/MC 30	17.5±2.0	3.4±0.5	0.74±0.04

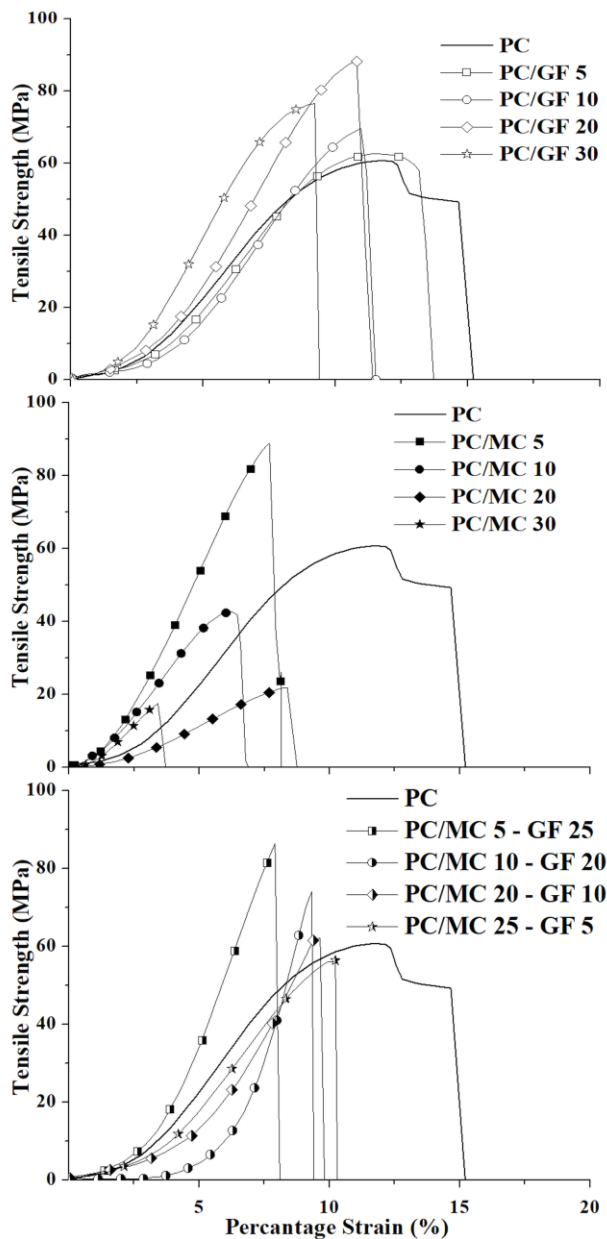


Fig. 1. Stress-strain curves of PC and composites.

According to **Table 1** the addition of 5% MC to PC shows about 50% improvement in tensile strength. As mica content increases, tensile strength decreases drastically. MC containing composites with 20% and 30% concentrations give the lowest strength values that may be stem from the agglomeration of high content mica particles. In the case of elongation parameters, MC addition causes about 50% reduction relative to unfilled PC. Tensile modulus results of PC/MC composites are similar with strength results that Young's modulus increases about 50% with the lowest MC loading. However, further addition of MC yields decreasing in modulus of composites remarkably which may be caused by the agglomeration of MC flakes [31,33] GF additions level up tensile strength of PC towards to 20% GF

containing composites. 30% GF filled PC composite yields reduction for tensile properties. This reduction may come from decreasing of fiber orientation by their high loadings [34-38].

Elongation of GF loaded composites display decreasing trend compared to PC. Tensile modulus of PC increases slightly with the GF additions up to concentration of 20%. As the GF content increases, elongation at break slightly decreases. Hybrid composites show nearly average values between individual GF and MC additions. Hybrid incorporations of MC and GF to PC cause sharp decreases in tensile strength. For hybrid composites, the highest tensile strength and modulus are obtained for minimum MC and maximum GF loadings (MC 5-GF 25). Strain values of hybrid composites also drop down with respect to unfilled PC.

### Impact resistance

Impact test is a high strain rate test that determines the amount of energy absorbed by a material during fracture. This absorbed energy is a measure of the toughness of a material and used to study the temperature dependent ductile/brittle transition. Impact test results of PC and its composites are shown in **Fig. 2**. Impact strength of PC exhibits reduction with GF loadings. The maximum impact strength among all composites is observed for the lowest mica content (MC 5). As the mica concentration increases impact strength decreases drastically. For the hybrid composites, the highest impact strength is observed for the PC/MC5 GF25 composite sample. The lowest mica loaded composite gives relatively higher results in the case of impact strength values. These findings are in accordance with the impact test results postulated in literature [39-43].

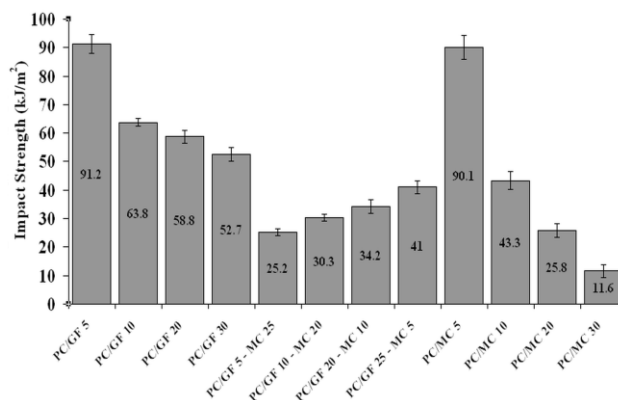


Fig. 2. Impact strength values of PC and composites.

### Hardness test

Shore D hardness values of PC and its composites are listed in **Table 2**. The hardness of PC gives sharp increase with CF concentrations. MC additions also cause improvement for hardness of unfilled PC. However, degree of increase is found to be lower for MC with respect to GF filled composites. In other words,

composites containing GF show slightly higher hardness values than that of MC at the same filling ratios. Composite loaded with 30% GF displays the highest value in which leads to about 4.3 units increase in Shore D hardness of unfilled PC. In the case of hybrid composites, hardness increases as the GF content increase. The highest hardness value is obtained for PC/MC5 GF25 among hybrid composites. The similar result is found from the literature that fiber loadings enhance Shore hardness of composites [44-46].

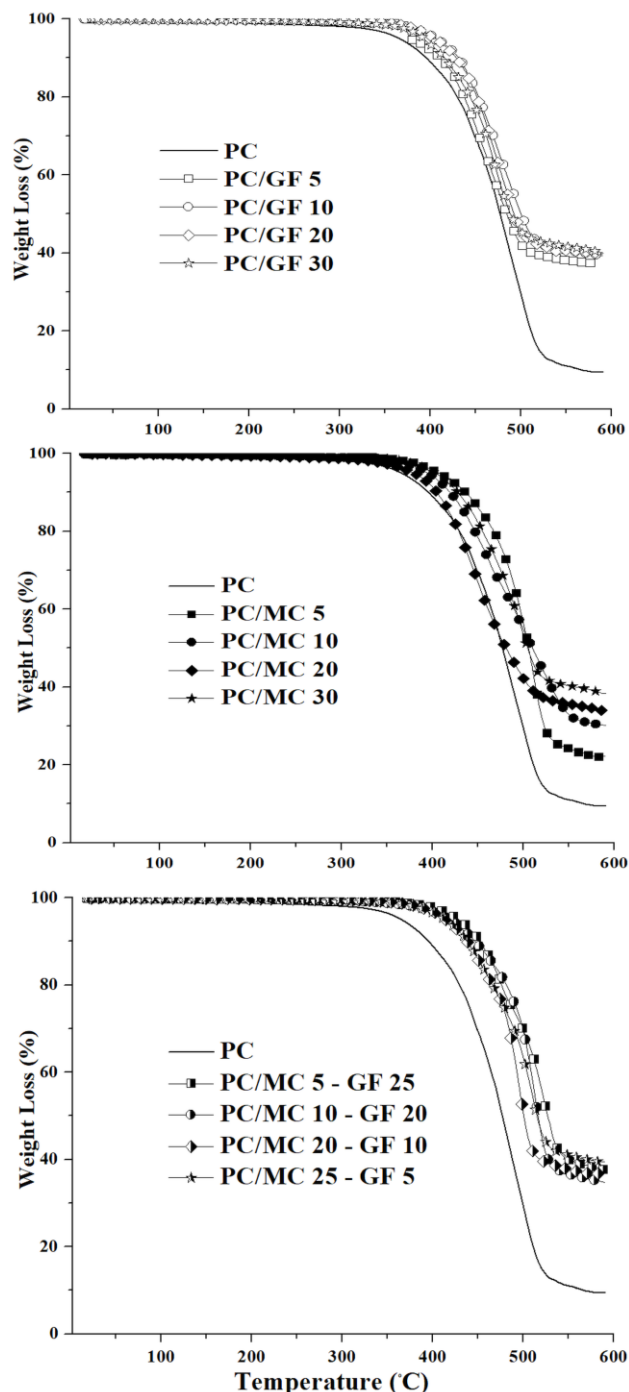
**Table 2.** Hardness values of PC and composites.

Samples	Hardness (Shore D)
PC	77.0±0.1
PC/GF 5	79.5±0.2
PC/GF 10	80.2±0.2
PC/GF 20	80.8±0.1
PC/GF 30	81.3±0.2
PC/MC 5 – GF 25	80.3±0.1
PC/MC 10 – GF 20	79.8±0.1
PC/MC 20 – GF 10	79.5±0.2
PC/MC 25 – GF 5	79.3±0.2
PC/MC 5	77.6±0.2
PC/MC 10	77.9±0.1
PC/MC 20	78.4±0.2
PC/MC 30	78.9±0.1

### TGA study

TGA test determines the decomposition rates of plastics and their corresponding temperatures. TGA curves are used to compare the thermal stabilities of polymers and their composites. The onset temperature for weight loss is considered as beginning of the thermal decomposition. Moreover, the amount of volatile matter and ash content can be found by the help of TGA graphs [47,48].

TGA curves of PC and relevant composites are shown in **Fig. 3**. Thermal decomposition of unfilled PC starts around 350°C due to degradation of carbonate, isopropyl and aromatic groups in the structure [49-51]. Between the temperatures of 400 and 600°C, significant weight loss values are observed for PC and composites. Additions of GF and MC shift the decomposition temperature of PC to higher levels. The higher degree of shifting occurs on thermo-grams of mica filled composites for their lower MC concentrations that is arising from the dispersion of plate-like geometry of MC particles [52-54]. Additionally, incorporation of hydrophilic filler contributes to hydrolytic degradation of PC during the melt-mixing stage. According to TGA curves of hybrid composites, thermal stability increases with the concentration of GF. PC/MC 5 – GF 25 and PC/MC 10 – GF 20 composites exhibit nearly identical decomposition temperature and these candidates have higher thermal stability among composites. GF containing composites yield much higher amount of char as compared to MC filled composites.



**Fig. 3.** TGA curves of PC and composites.

### DSC analysis

Glass transition temperatures ( $T_g$ ) of PC and composites that estimated from DSC analysis data are listed in Table 3. It can be seen from **Table 3** that  $T_g$  of the 5% MC containing composite is found to be slightly higher with respect to  $T_g$  of PC, it shifts to lower temperatures with the further addition of MC. The inclusion of GF in PC causes improvement for  $T_g$  of pure PC. This increasing trend is observed from 5% to 20% GF concentrations, then sharp

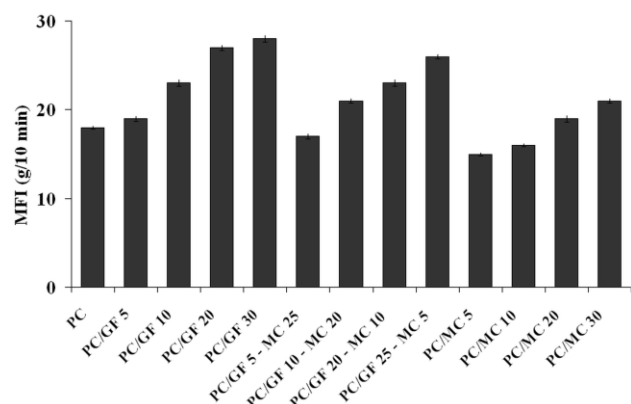
decrease is taken place for PC/30 GF composite.  $T_g$  of hybrid composites are almost identical at all of the compositions investigated. Hybrid composites give slightly lower  $T_g$  values compared to GF filled composites. The additions of MC and GF reduce the percent crystallinity as the amount of additives increases which may be stem from the restriction in segmental rearrangements of PC chains [55].

**Table 3.** DSC test results of PC and composites.

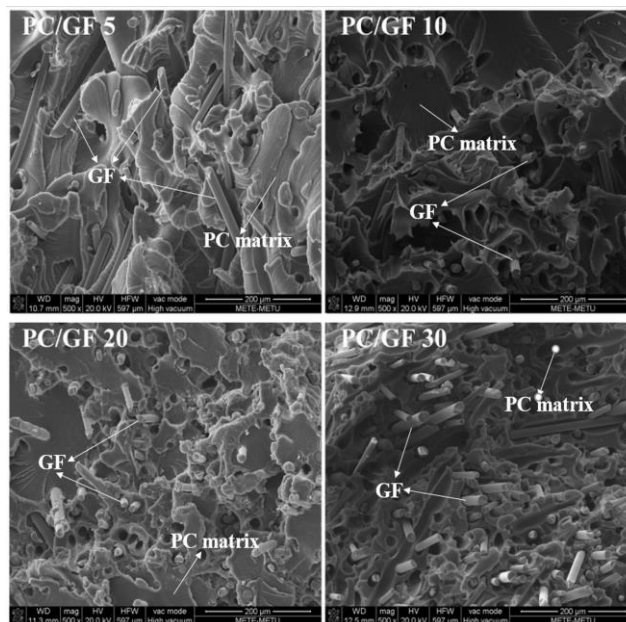
Samples	$T_g$ (°C)
PC	141.53
PC/GF 5	142.29
PC/GF 10	142.64
PC/GF 20	143.01
PC/GF 30	140.75
PC/MC 5 – GF 25	141.04
PC/MC 10 – GF 20	141.57
PC/MC 20 – GF 10	141.41
PC/MC 25 – GF 5	141.77
PC/MC 5	142.41
PC/MC 10	141.68
PC/MC 20	141.55
PC/MC 30	141.80

#### MFI measurements

As represented in **Fig. 4**, MFI results show that the additions of GF cause improvement for MFI of PC. This increasing trend is observed obviously for higher GF loaded composites. Hydrolytic degradation of PC during the melt processing may led to reduction for MFI values [56]. The MFI value decreases with addition of 5% and 10% of MC to PC matrix. However, the higher level of MC contents displays increasing trend in MFI values. Observation of lower MFI values for PC/MC composites relative to PC/GF ones may be related with layered structure of mica particles which have larger surface area [57-60]. In the case of hybrid composites, MFI results were almost averages of PC/GF and PC/MC composites. For the hybrid composites, the MFI value increased with GF inclusions and decreased with mica additions due to aspect ratio of GF [61,62].



**Fig. 4.** MFI values of PC and composites.



**Fig. 5.** SEM micrographs of PC/GF composites.

#### Morphology of composites

Morphological investigations of composites were performed using SEM micrographs of composites. Distributions of added fillers were examined with the help of SEM images taken from the fractured surfaces of composites. Pull-out of fibers yield dark holes as can be seen from SEM micrographs of PC/GF composites. According to **Fig. 5**, fibers are homogeneously dispersed into PC phase for the lowest GF containing composite. As the concentration of GF increase, bundle formations of fibers are observed. Individual fibers can be seen as detached from PC matrix due to poor adhesion between two phases for high loading level of GF. The distribution of GF in PC matrix is found to be more homogeneous for PC/GF20 than PC/GF30. The distributions of GF for given percentages are in agreement with mechanical tests results discussed in earlier sections in which 20% concentration is remarked as the optimum value for GF loaded PC composites. **Fig. 6** displays the SEM micro-images of MC filled composites. Mica particles exhibit the tendency of adhesion to each other rather than the interaction with polymer matrix. This observation may also be caused from the inert surface of mica for PC that results with incompatibility. The investigation of scanning electron micrographs of hybrid composites in **Fig. 7** shows that the most homogenous distribution is observed for 5% mica and 25% GF filled PC composites. The agglomerations are seen dominantly as mica content increase, especially for 20% and 30% MC containing hybrid composites. The lowest mica loaded composite (5%) display homogeneous distribution of mica particles into PC matrix. However, as the mica content increase agglomerations start to dominate in morphology of composites.

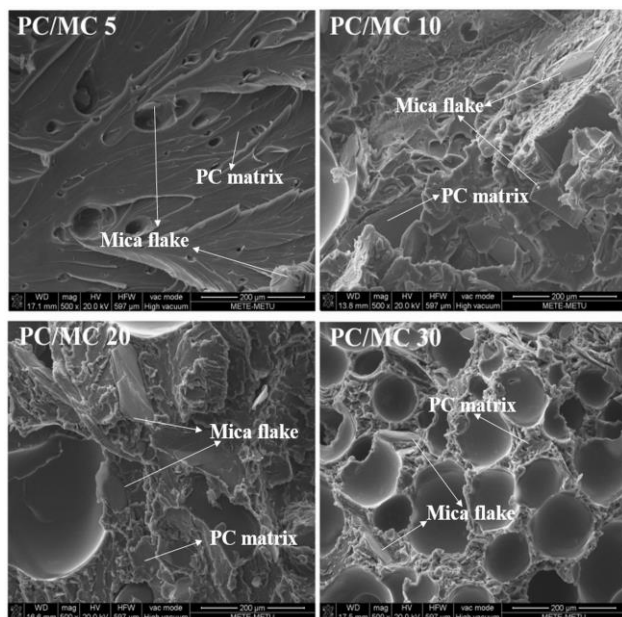


Fig. 6. SEM micrographs of PC/MC composites.

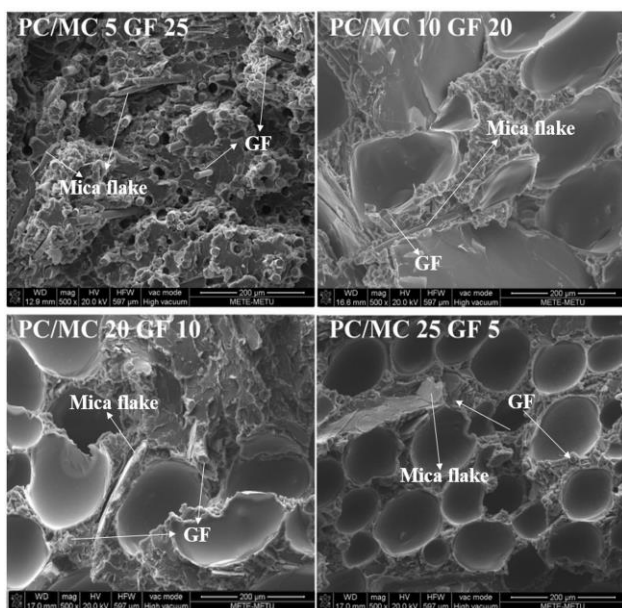


Fig. 7. SEM micrographs of hybrid composites.

## Conclusion

In this study, glass fiber and mica were added to polycarbonate matrix as binary and ternary in hybrid form. Processing methods were chosen as melt mixing and injection molding for the purpose of practical adaptation to large scale composite applications in mainly transportation and construction fields. The mechanical, flow, thermal and morphological characterizations of produced composites were examined via tensile, hardness and impact test, MFI measurements, TGA and DSC analyses and SEM techniques, respectively. The overall results revealed that the optimum values for mica containing, glass fiber reinforced and hybrid forms were

found to be 5%, 20% and 25% GF-5% MC filling ratios, respectively. The maximum tensile test values (tensile strength and modulus), impact strength and glass transition temperatures estimated from DSC were obtained in these optimum concentrations of composites. The further additions of these fillers caused agglomerations for mica particles and bundle formations for glass fibers according to SEM micrographs. Such formations caused restriction of their homogeneous distribution in the PC matrix. MFI test results revealed that MC filled composites gave lower MFI values compared to the GF containing ones which attributed to the higher surface area of mica flakes. There was no synergy observed between MC and GF because of their geometrical differences. For this reason, individual additions of GF and MC resulted in higher mechanical and thermal performance compared to hybrid composites.

## Keywords

Polycarbonate, glass fiber, mica, extrusion, hybrid composites.

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