






Improving the mechanical and thermal properties of chlorinated poly(vinyl chloride) by incorporating modified CaCO₃ nanoparticles as a filler

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Abstract: Chlorinated poly(vinyl chloride) (CPVC)/calcium carbonate nanocomposites were successfully prepared by the incorporation of calcium carbonate (CaCO₃) nanoparticles into the CPVC matrix. The compatibility between the two phases was obtained by surface modification of the CaCO₃ nanoparticles with stearic acid, leading to improved material performance. The effects of the addition of different amounts of CaCO₃ nanoparticles to the CPVC on the thermal, mechanical, and morphological characteristics of the CPVC/CaCO₃ nanocomposites were investigated. The thermal stability of the CPVC/CaCO₃ nanocomposites was evaluated by thermogravimetric analysis and differential scanning calorimetry. In addition, the surface texture of the CPVC and the dispersion of the CaCO₃ were evaluated using scanning electron microscopy. Important enhancements in the thermal and mechanical properties of the modified CPVC/CaCO₃ nanocomposites were obtained by incorporating different amounts (2.00%, 3.75%, and 5.75%) of surface-modified CaCO₃ nanoparticles within the CPVC polymer matrix. The results reveal that 3.75% of CaCO₃ was the optimum amount, where the CPVC/CaCO₃ nanocomposite shows the highest impact strength, the highest tensile strength, the highest thermal stability, and the lowest elongation percentage. Replacement of the commercial impact modifier used in industry with the prepared surface-modified CaCO₃ nanoparticles for the development of CPVC was successfully achieved.

Key words: Chlorinated poly(vinyl chloride), CaCO₃ nanoparticles, nanocomposites, mechanical properties, thermal properties

1. Introduction

Polymer nanocomposites are considered a hot research area due to their extraordinary properties and distinctive design feasibility over pristine polymers. The properties of polymer-based nanocomposites depend upon many parameters such as the shape, chemical and physical characteristics, and the amount of constituent (filler) and interfacial interactions between the two phases [1–3]. Nanocomposites often exhibit properties that are significantly different from those of traditional composites in which a micrometer level inorganic component is

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added to a polymer matrix [4–6]. By modifying the properties of the filler, adhesion between the matrix and filler can be enhanced.

Calcium carbonate is a versatile mineral and known as an essential ingredient in the paper, paints, coatings, rubbers, adhesives, sealants, and plastics industries. Calcium carbonate particles have been added to plastics to reduce costs [7–9]. Certain properties such as the thermal, mechanical, and rheological properties of the resulting plastics were improved by CaCO_3 addition [10–12]. CaCO_3 nanoparticles have already been used as fillers in thermoplastics and especially in poly(vinyl chloride) (PVC) to improve some properties, such as mechanical and dimensional stability, gas barrier, heat resistance, and flame retardancy [13]. Surface modification of CaCO_3 nanoparticles is recognized as a useful processing aid as an impact modifier for rigid PVC [14]. Surface-modified CaCO_3 nanoparticles, which are more easily dispersed uniformly in a polymer matrix, provided various benefits to the PVC matrix [15,16].

The compounding of PVC with surface-modified CaCO_3 nanoparticles significantly improved the surface gloss and the processability of the PVC during injection molding [17,18]. Moreover, the damage to extruder barrels and screws was minimized if the CaCO_3 nanoparticles were coated with a fatty acid. Furthermore, surface-modified CaCO_3 nanoparticles reduce the gelation time of PVC as they have better compatibility with PVC primary particles. In addition, surface-modified CaCO_3 nanoparticles can stabilize the dispersion of the components of PVC to eliminate the plate-out problem. More importantly, surface-modified CaCO_3 nanoparticles lead to improvements in both the impact strength and the stiffness strength of the polymer [19–21].

CPVC characterized with high thermal and chemical stability was used in the production of pipes such as those made of high density polyethylene (HDPE). A comparison study was carried out between a CPVC pipe and one made from HDPE [22]. The results showed that HDPE is a powerful ductile material, but CPVC is characterized as a robust material withstanding very high pressure and temperature despite its fragile behavior. In addition, CPVC can be used in different applications, such as in hot and cold water plumbing distribution, as well as in fire protection in manufactured homes and light hazard occupancies, reclaimed water piping, and chilled piping and hydronic piping and distribution (radiators, fan coils). Moreover, it can be used in mining, aerospace, high tension cable protection, solar heating, and radiant floor heating [23,24].

In the present study, CaCO_3 nanoparticles with a definite particle size (0.1 μm) were incorporated in different amounts in CPVC polymer to study the improvements in the properties of CPVC base nanocomposites. The objectives of the present work were: (i) to replace the commercial impact modifier used in industry by surface-modified CaCO_3 nanoparticles for the development of chlorinated PVC (CPVC)/ CaCO_3 nanocomposites and (ii) to improve the mechanical, thermal, and morphological properties of the CPVC/ CaCO_3 nanocomposites. Therefore, different amounts of surface-modified CaCO_3 nanoparticles (2.00%, 3.75%, and 5.75%, based on EDX analysis) were added to the CPVC polymer matrix. The mechanical, thermal, and morphological properties were then investigated.

2. Results and discussion

2.1. The mechanical properties

Figure 1 shows the impact strength data of the CPVC/ CaCO_3 nanocomposites. It can be seen that the impact strength value increased when different amounts of surface-modified CaCO_3 nanoparticles were added to the CPVC matrix instead of commercial impact modifier (PMMA). Moreover, the optimum impact strength value

($\approx 160 \text{ kJ/m}^2$) was observed for the nanocomposite with the composition of PMMA 92%/CaCO₃ 3.75%. With further increment of CaCO₃ content (5.75%) the impact strength value decreased. It was thought to be due to the formation of CaCO₃ aggregates, which affected the homogeneous distribution of CaCO₃ nanoparticles within the CPVC matrix.

Figure 2 displays the effect of the addition of different loading amounts of CaCO₃ nanoparticles (2.00%, 3.75%, and 5.75%) to CPVC in comparison with the 100% PMMA on the tensile strength of the prepared nanocomposite. As shown in Figure 2, the tensile strength slightly increased with increasing CaCO₃ content to reach the optimum value of $\approx 577 \text{ kg/cm}^2$ at 3.75%. Then the tensile strength value decreased at higher amounts of CaCO₃ due to the formation of CaCO₃ aggregates within the CPVC polymer matrix. Moreover, the tensile strength obtained for 5.75% was 549 kg/cm^2 , which is greater than the value obtained with the CPVC composite with the acrylic impact modifier. It can be concluded that the addition of the surface-modified CaCO₃ nanoparticles to the CPVC polymer improved the impact and the tensile strength compared to the commercial impact modifiers [12]. The improvements are correlated with the toughness and the stiffness of the nanocomposites.

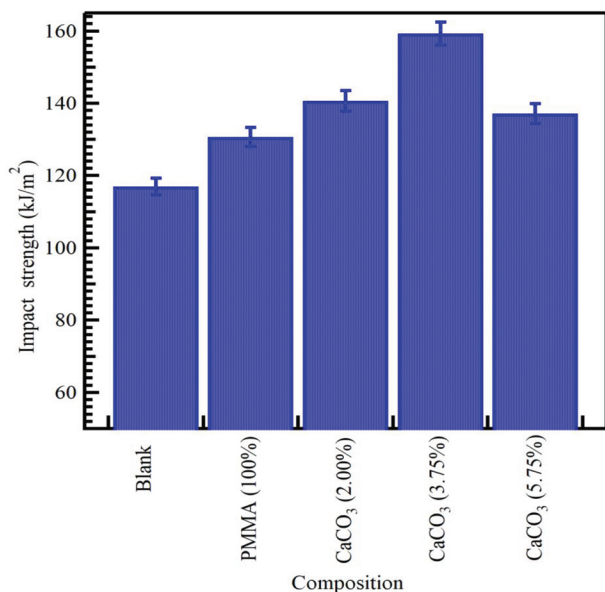


Figure 1. Variation in the impact strength of CPVC/CaCO₃ nanocomposites with CaCO₃ content.

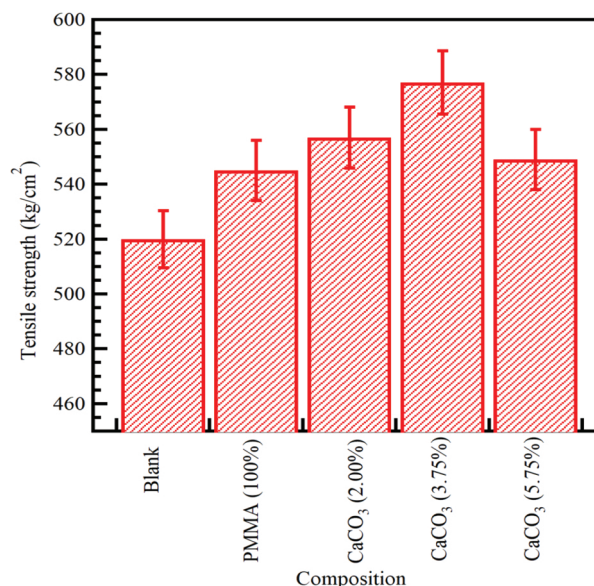


Figure 2. Variation in the tensile strength of CPVC/CaCO₃ nanocomposites with CaCO₃ content.

Figure 3 displays the effect of the loading amounts of modified CaCO₃ nanoparticles on the elongation (%) of the CPVC/CaCO₃ nanocomposites. The elongation was found to be decreased with increasing amount of the modified CaCO₃ nanoparticles in comparison with the acrylic impact modifier. This indicates that the presence of acrylic impact modifiers (PMMA 100%) provides high elongation compared with blank CPVC and CPVC/CaCO₃ nanocomposite with different amounts of CaCO₃ nanoparticles. That may be explained as follows: by increasing the amounts of CaCO₃, the rigidity of the prepared nanocomposites is increased. Then the elongation decreased with the increase in the CaCO₃ nanoparticles, which was the opposite of what happened to other mechanical properties (impact strength and tensile strength) as mentioned above. This is due to the interface debonding/voiding and matrix deformation caused by CaCO₃ nanoparticles, which act as stress concentrators. In addition, CaCO₃ nanoparticles have a hydrophilic surface with high surface energy, which

generates low compatibility with the hydrophobic polymer matrix. It has been reported that some mechanical properties of the composite such as the elongation at break point were rather poor as the CaCO_3 content was increased [11,25].

Vicat softening point (VSP) is a measure of the softening point of materials that have no definite melting point. Here Vicat results were used to compare the heat characteristics of different prepared CPVC/ CaCO_3 nanocomposites. As shown in Figure 4, the presence of acrylic impact modifier resulted in a VSP value slightly higher than that of blank (pristine CPVC); however, the VSP increased from 111.0 to 112.5 °C for CPVC/ CaCO_3 nanocomposites in comparison to blank CPVC. It was found that the VSP increased with increasing CaCO_3 content as impact modifier up to 3.75%, followed by a decrease at higher loading values.

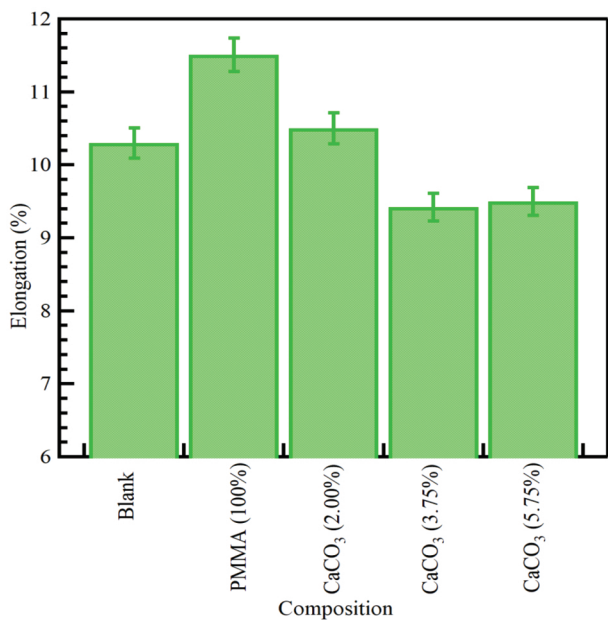


Figure 3. Variation in the elongation of CPVC/ CaCO_3 nanocomposites with CaCO_3 content.

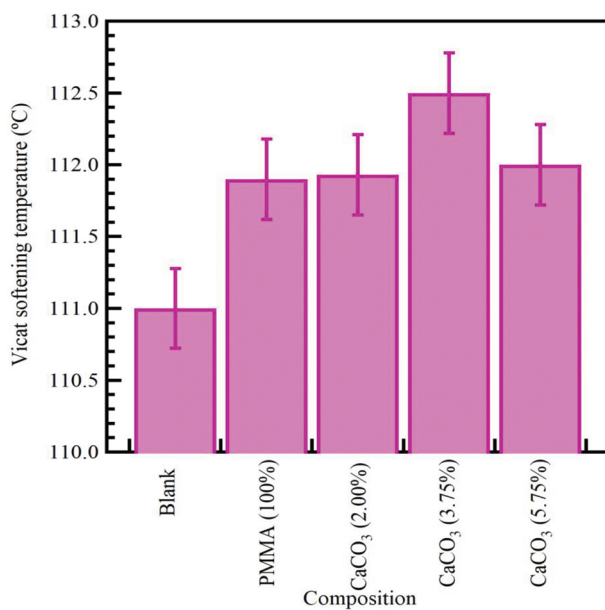


Figure 4. Variation in vicat softening temperature of CPVC/ CaCO_3 nanocomposites with CaCO_3 content.

2.2. Thermal properties

Figure 5 shows the TGA curves of pure CPVC polymer and CPVC/ CaCO_3 nanocomposites containing different amounts of modified CaCO_3 nanoparticles. The addition of modified CaCO_3 nanoparticles to the CPVC matrix improved the thermal stability of the prepared nanocomposites when compared with the pure CPVC polymer. Moreover, the CPVC/ CaCO_3 nanocomposite containing 3.75% CaCO_3 was the most stable sample among pure CPVC and other nanocomposites. Moreover, the TGA curves showed that there is a common thermal degradation stage with all the investigated samples. This weight-loss stage is observed within the temperature range of 150–380 °C and is mostly due to the HCl elimination from CPVC polymer and the formation of hydrocarbon products such as polyene structures. Finally, it can be concluded that addition of the modified CaCO_3 nanoparticles improved the thermal stability of the nanocomposites compared with the blank CPVC polymer matrix [26].

Figure 6 represents the observed glass transition temperature, which was determined by the DSC curves, for pure CPVC and CPVC/ CaCO_3 nanocomposites containing different amounts of modified CaCO_3 nanoparticles. The glass transition temperature (T_g) of the blank CPVC polymer was around 110.3 °C, and those

containing 2.00%, 3.75%, and 5.75% CaCO_3 were 98.39, 70.60, and 63.11 °C, respectively. The decrease in the T_g of the polymer nanocomposites could be due to the surface modification of nano- CaCO_3 , where it works as a lubricant for the polymer chains. This means that by increasing the amount of the modified CaCO_3 nanoparticles the glass transition temperature became lower. The difference in the glass transition temperature was correlated with the increase in the glassy behavior and rigidity of the CPVC/ CaCO_3 nanocomposites.

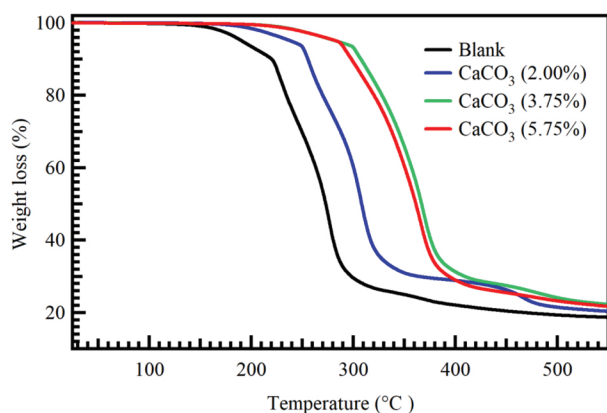


Figure 5. TGA curves for the pure CPVC and CPVC/ CaCO_3 nanocomposites.

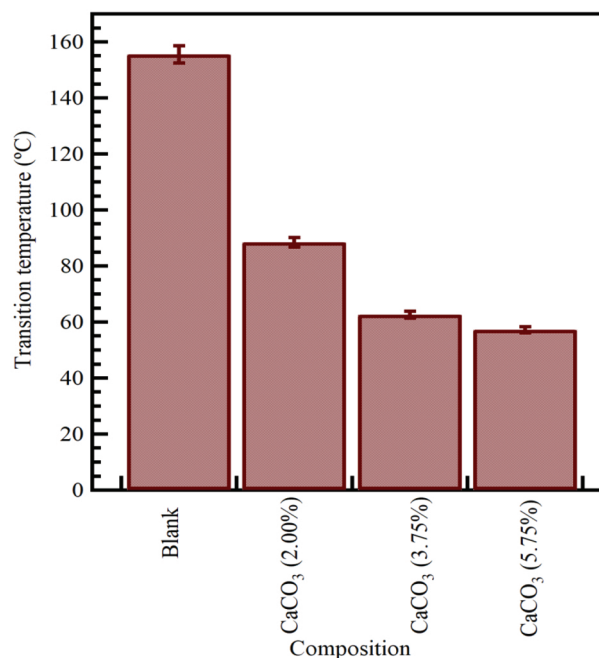


Figure 6. Glass transition temperature for the pure CPVC and CPVC/ CaCO_3 nanocomposites.

Figure 7 represents the SEM images of the fractured surface of pure CPVC polymer (Figure 7a) and CPVC/ CaCO_3 nanocomposites (Figures 7b–7d). It is clearly seen that pure CPVC shows a single phase structure, while in the case of the modified CaCO_3 nanoparticles fine and homogeneous nanoparticles are observed in the CPVC matrix [27]. The amount of dispersed nanoparticles increased with increasing loading ratio, and Figure 7d shows that a higher amount of modified CaCO_3 (5.75%) is dispersed over the surface of the CPVC matrix compared with other low CaCO_3 content nanocomposites (2.00% and 3.75%). Despite the presence of stearic acid to prevent the nanoparticles leaving the polymer matrix, it was not possible to prevent the agglomeration of CaCO_3 nanoparticles at higher loading. The SEM analysis confirmed the presence and the dispersion of the modified CaCO_3 nanoparticles within the CPVC polymer matrix and the size of the dispersed CaCO_3 nanoparticles (65–135 nm) within the CPVC polymer matrix. In addition, EDX analysis was performed to obtain more information on the samples' elemental composition. Figure 8 shows the EDX data of pure CPVC and the nanocomposites with different amounts of CaCO_3 (2.00%, 3.75%, and 5.75%). The corresponding elemental ratios are shown in the insets. The pure CPVC shows the presence of C, Cl, and O with elemental ratios of 64.4%, 26.2%, and 9.4%, respectively. Moreover, all nanocomposites show the presence of Ca as a result of CaCO_3 addition, which is totally absent in the pure CPVC. The ratio of Ca was 0.8%, 1.5%, and 2.3%, which is equivalent to 2.00%, 3.75%, and 5.75% CaCO_3 content. The EDX results are in agreement with the SEM observation.

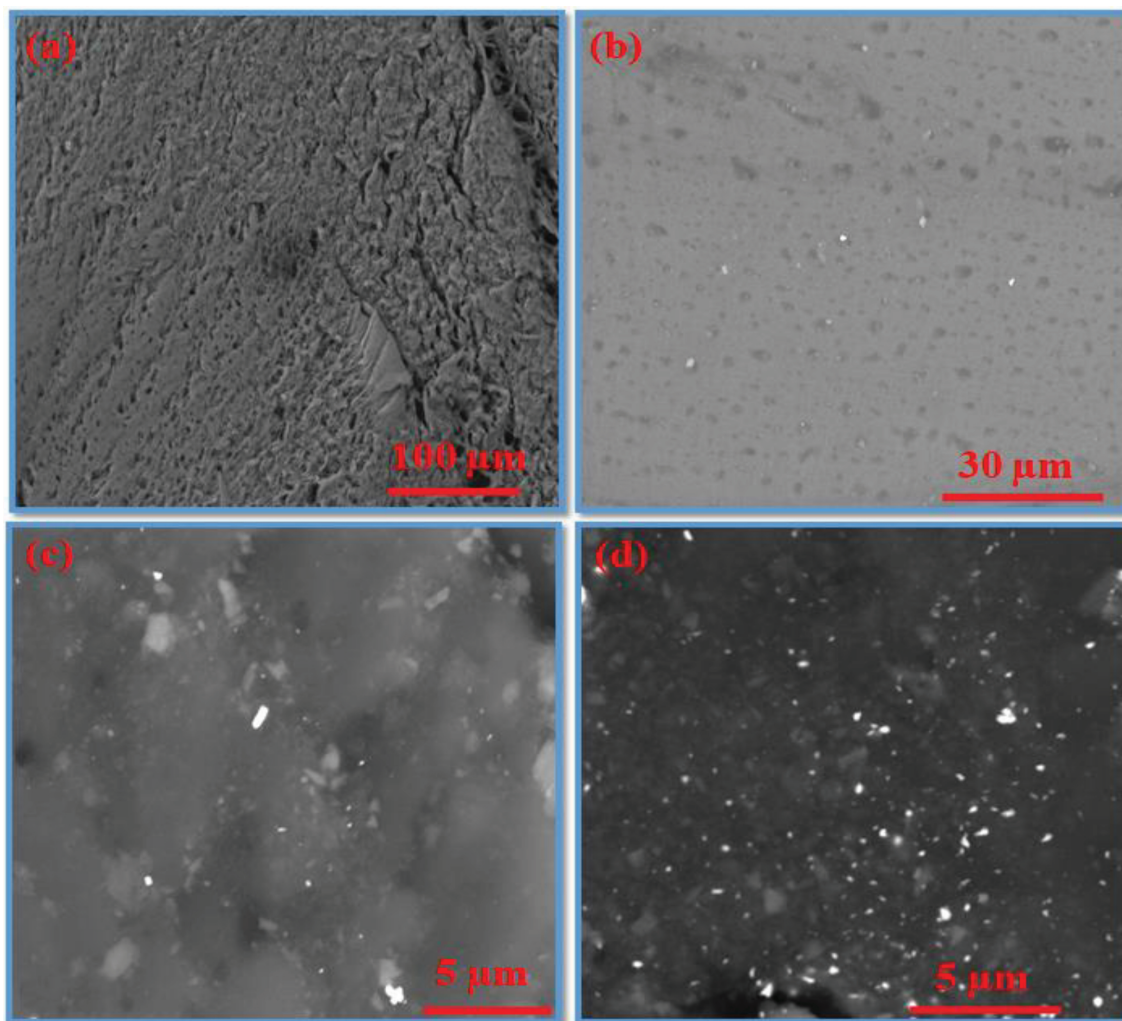


Figure 7. SEM micrographs of a) pure CPVC, b) 2.00% CaCO_3 , c) 3.75% CaCO_3 , and d) 5.75% CaCO_3 .

2.3. Conclusions

CPVC/ CaCO_3 nanocomposites with different amounts of surface-modified CaCO_3 nanoparticles were successfully prepared. The thermal, mechanical, and morphological properties of the CPVC/ CaCO_3 nanocomposites were examined and compared with those of pristine CPVC and CPVC-based composite with a commercial acrylic impact modifier. CPVC's thermal stability and mechanical properties were significantly enhanced by complexation with the modified CaCO_3 nanoparticles as a result of replacement of the acrylic impact modifiers with the prepared surface-modified CaCO_3 nanoparticles. Thus, surface-modified CaCO_3 nanoparticles are recommended as a suitable impact modifier and additive for CPVC resin that is cheaper than the commercial one.

3. Materials and methods

3.1. Materials

CPVC suspension and calcium carbonate (FC500) were purchased from Oxy Vinyls LP (Dallas, TX, USA) and the Formosa Plastics Corporation (Taipei, Taiwan), respectively. CaCO_3 nanoparticles (SP-200, surface

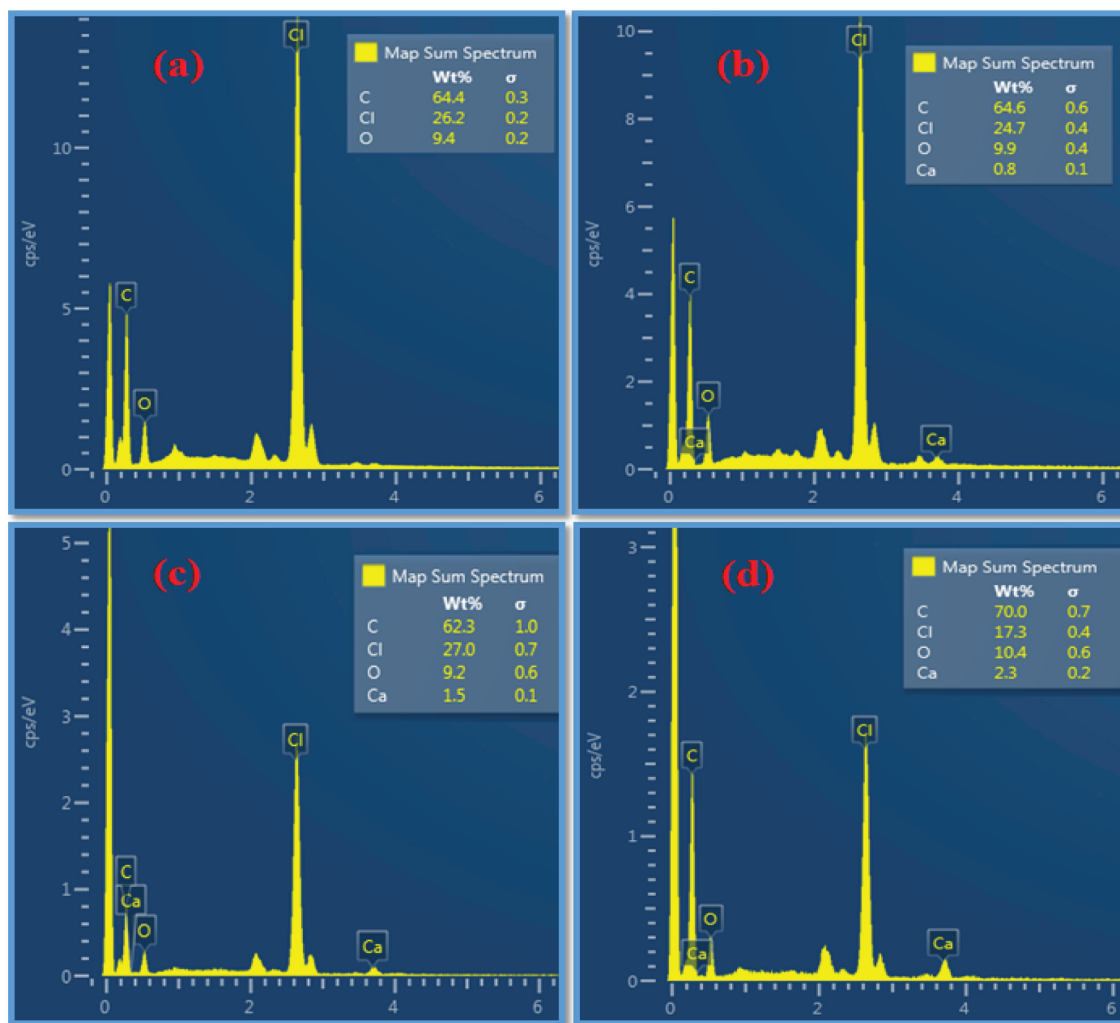


Figure 8. EDX analyses of a) pure CPVC, b) 2.00% CaCO₃, c) 3.75% CaCO₃, and d) 5.75% CaCO₃.

modified with stearic acid) and titanium dioxide were produced by Shanghai Perfection Nanometer New Material Co. Ltd. (China) and KORONOS (Montreal, Canada), respectively. Calcium stearate was obtained from Zirve Polymer (İstanbul, Turkey). Processing aid and tin stabilizer were purchased from LG Chem. (USA) and Galata Chemicals (Germany), respectively. Tin was used as a thermal stabilizer. Polyethylene wax (PED521) was purchased from Clariant International Ltd. and soya bean oil was obtained from INBRFLEX A-6 FRP Services (New York, NY, USA); both materials were used as lubricating agents to decrease the fraction process inside the extruder.

3.2. Method

3.2.1. Surface modification of CaCO₃ nanoparticles

CaCO₃ nanoparticles placed in a high speed mixer and heated up to 105 °C. Then the stearic acid was added. The temperature of the mixer was raised to 125 °C and then cooled to room temperature at a rate of 2 °C/min.

3.2.2. Preparation of PMMA/CaCO₃ nanocomposite as an impact modifier

The polymerization of MMA monomer was effected in the presence of 10% CaCO₃ nanoparticles by in situ emulsion polymerization at 70 °C. This in situ polymerization was performed by batch seed emulsion polymerization in a 500-mL glass reactor (stirring rate 200 rpm) as follows. First, nano-CaCO₃ was dispersed in aqueous solution ultrasonically. The ingredients (deionized water, sodium dodecyl sulfonate as emulsifier, and MMA as monomer) were added to the reactor vessel and the resulting mixture was heated and stirred continuously until the reaction temperature was reached. Then an aqueous solution of potassium persulfate with 1% MMA as initiator (preheated to reaction temperature) was added to start the polymerization reaction and the reaction time was recorded. The glass reactor equipped with a stirrer was purged with N₂ gas six times. The products were spray dried at 80 °C after emulsion breakage. Subsequently, they were dried at 60 °C under vacuum until constant weight for 48 h.

3.2.3. Polymer nanocomposite preparations

A high-speed mixer was used to premix CPVC 100 phr, with tin stabilizer 5 phr and stearic acid 0.3 phr for 8 min to give compounded CPVC as given in the Table. The CaCO₃ nanoparticles were dried at 70 °C for 48 h under vacuum. The CPVC matrix and the CaCO₃ nanoparticles were melt mixed in a two-roll mixer at 140 °C for 15 min to obtain a binary nanocomposite. CPVC and the surface-modified CaCO₃ nanoparticles were mixed in a similar way to prepare CPVC/CaCO₃ nanocomposites with different compositions. Sheets with a testing thickness of 1 and 3 mm were prepared by compression molding at T = 150 °C and P = 20 MPa pressure for 10 min, and then cooled to room temperature at P = 10 MPa. A series of the nanocomposites were prepared in which the amount of CaCO₃ was varied from 2.00 to 5.75 wt.%. Based on the literature survey, the optimal amount of the filler was found to be 2.5%–5% [28]. On the other hand, by increasing the amount of the nanoparticles (15–20 wt.%), the chance of particle agglomeration will increase due to the inherent thermodynamic drive of nanoparticles [29]. Therefore, this study range of the filler content in this work is considered the optimum.

Table. The formulation of the CPVC nanocomposites.

Material	Amount (phr)	Material	Amount (phr)
CPVC resin	100	Processing aid (acrylic acid)	1.7
Titanium dioxide	5.8	Tin stabilizer	4.2
Polyethylene wax	0.125	Soya bean	2.1
Calcium stearate	0.625	CaCO ₃ nanoparticles	4, 8, and 12

3.3. Characterization techniques

The tensile strength and elongation measurements were conducted at a crosshead speed of 50 mm/min using a Zwick tensile tester according to ASTM D638M. A thermogravimetric analyzer (Shimadzu-TGA 50) was used to study the thermal stability using about 20 mg of the samples at a heating rate of 10 °C/min under inert atmosphere. A differential scanning calorimeter (Shimadzu-DSC 50H thermal analysis systems, Japan) was used for the determination of the glass transition temperature (T_g). The surface morphology of the composites was observed by scanning electron microscopy (SEM), with an EDX analyzer using a HITACHI-S-2150 apparatus.

Acknowledgments

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