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Synthesis and Characterization of Polyvinyl Chloride Doped Tellurium Compound

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Abstract

In this study, Chloride doped Tellurium Compound (PVC/Te) was synthesized chemically in an alkaline medium under nitrogen to remove and avoid any oxidation. Fourier transform infrared (FTIR), X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray analysis (EDX), and Thermogravimetric analysis (TGA) techniques are used to characterize it. XRD pattern showed a more degree of crystallinity for PVC/Te compared with behaviour for pure PVC. The molecules are regularly arranged for the effect by tellurium-Carbon bonds. Moreover, the average crystallite size of 40.47nm is obtained from Deby-Scherrer equation calculations. The ratio of Te in PVC/Te according to EDX is 16% weight of Tellurium. The FESEM technique revealed that PVC/Te uniform decreased as the electron beam energy increased. In addition, TGA results were used to evaluate the thermal stability of compounds, where the Polyvinyl Chloride Doped Tellurium Compound was more thermal stable than pure PVC.

Introduction

Despite its importance in industry, PVC has some drawbacks including poor thermal stability to heat, ultraviolet light due to dehydrochlorination [1]. Low mechanical reinforcement [2] and deficiency in other physical properties such as electrical and optical properties [3]. As a result, a considerable effort has gone into improving the performance and properties of PVC by combining various materials such as layered clay [4]. Inorganic particles, and other materials such as silica [5]. Calcium carbonate [6]. Potassium Bromide, [7]. Metalloids [8]. Transition elements, [9-12] and silane. The addition of such materials to polymers can result in new properties and, consequently, in new and exciting applications [13]. Adding an amount of fillers can increase the hardness of PVC composite due to their much higher elasticity modulus than that in the

PVC polymer. The mechanical properties of the matrix can also be enhanced resulting in a higher ability to transfer stresses from the polymer matrix to the embedded particles [14-18]. Meanwhile, the thermal coefficient of PVC expansion decreases with the addition of silica [19]. PVC/ZnO can upgrade electrochemical anticorrosive residences [20]. Incorporating 6-aminopyrimidine derivatives into the PVC backbone chains improved the samples' antimicrobial properties against bacteria and fungi [21].

Experimental procedure

1. Materials

PVC and pure Te were used as Composites. All substances were supplied by Aldrich, 20% sodium hydroxide NaOH, sodium borohydrides $NaBH_4$, Cyclohexanone as solvent for PVC.

2. Synthesis of the PVC/Te

The PVC/Te solution was synthesized by mixing 0.0033 mol of tellurium powder with 0.0132 mol of sodium borohydride in an alkaline medium (20% NaOH) under nitrogen and heat at 100°C in a three-necked flask until the solution became yellow. Color changes to reddish; after the reddish color had disappeared, the previously prepared PVC solution was added directly, dissolving 0.5 g PVC powder in 25 mL cyclohexanone. Cloud layers of aqueous, upper, organic, and lower stratum, for example, were separated. The pale-yellow filter is poured into a Petri dish until the solvent evaporates. The dried substance was then well crystallized and crushed with a mortar and pestle to study the composition and characterization of its components.

3. Characterization Techniques

The Fourier Transform Infrared (FTIR) spectra where ecorded in a range of 4000-400 cm-1 on a Shimadzu spectrometer using KBr discs. To study the crystalline structure by using Leakage X-Rays (Model: XRD-6000), diffractometer X-ray Tube (Cu 1.54059 Å), voltage: 40.0 kV, current: 30.0 mA, the spectra recorded a scan range of 10.000 - 90.000 deg. The morphology of the surface was studied by using FESEM at various gradually rising electronic energies and high magnification. In addition to employing EDX technology to calculate the percentage of elements in the manufactured compound. A study of the thermal behaviour was achieved by the TGA technique..

Results and discussion

1. Infrared spectroscopy

Figure 1. shows the FTIR spectra of the polyvinyl chloride PVC and PVC grafted Tellurium, where C–H stretching vibrations are responsible for the absorption peak observed at 2920 cm-1. The bands at 1325 cm-1 and 1425 cm-1 are the results of the C–H deformation of CHCl, and wagging of the CH2 deformation, respectively.22 The peak at 960 cm-1 represented C–H wagging vibration and CH2 rocking, while the peak at 1097 cm-1 described the C–C stretch vibration. The band at 690 cm-1was associated with the stretching vibration of C–Cl [23] , C–H bending vibrations originating from the PVC polymer's CHCl groups, and C-H rocking which were attributed to the peak at 1246 cm-1 [24].

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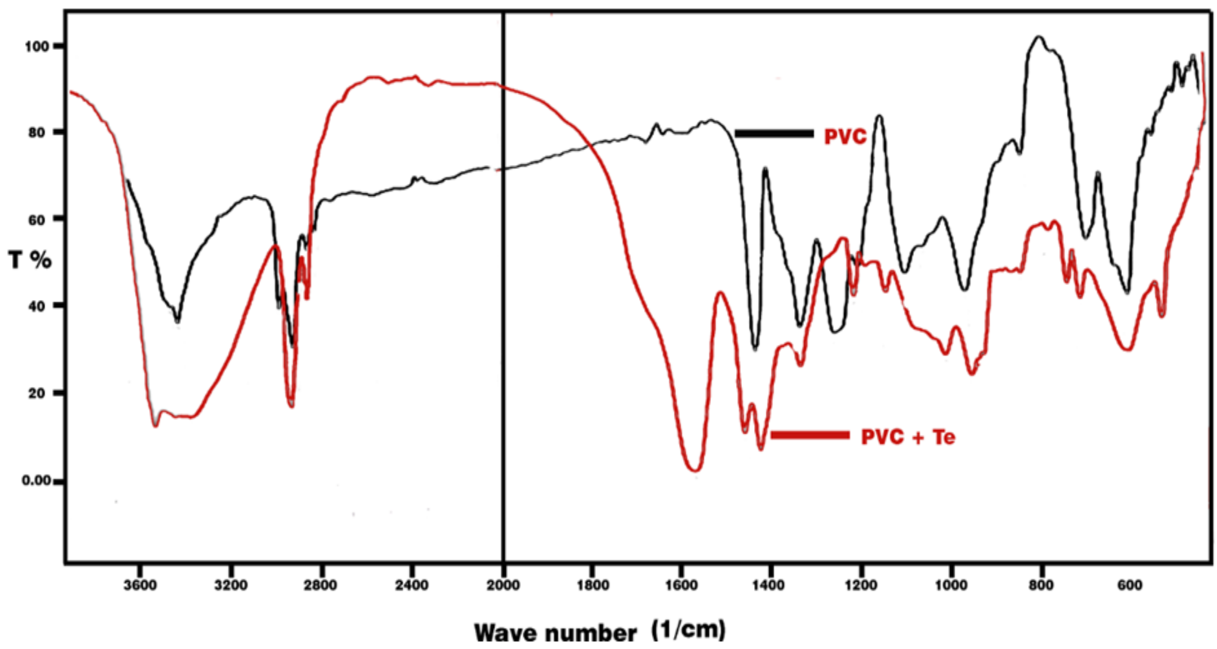

Figure 1: The FTIR spectra of Commercial PVC represented by the black line while the red line represented PVC/Te.

2. XRD

Figure 2 depicts the X-ray diffraction PVC and PVC/Te powder used in this study. The X-ray powder diffraction to characterize a solid-state sample is a powerful tool; the X-ray diffraction pattern can be used to determine the unknown species or to characterize the atomic-scale structure of a previously identified substance. Meanwhile, the XRD pattern appears to contain high-intensity Bragg diffraction peaks at 2θ = 23.03, 29.4,35.9, 42, 46, and 47. These are typical polyvinyl chloride XRD peaks [25]. The positions of these peaks are compared to the JCPDS card compound and its constituent elements. The patterns confirmed the amorphous nature of commercial PVC and its distinctive broad feature at 2θ 23 [26].The samples doped with Tellurium showed the crystallization phase PVC/Te with crystalline peaks at 2θ 30◦, 31◦, and 33◦ these peaks well match the (111), (100), and (001) crystal planes.

Table 1: XRD Parameters of PVC/Te

Peak No.	2Θ(deg)	d (A)	I/II	FWHM (deg)	Intensity (counts)	Integratedint (counts)
1	30.6754	2.9122	100	0.70930	778	2915
2	33.3357	2.6856	88	0.50000	685	2003
3	31.8171	2.8026	81	0.55500	629	2107

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The crystallite sizes of PVC/Te are calculated by Deby-Scherrer equation 1. Where the average crystallite size of 40.47nm.

$$D= K\lambda / \beta D \cos\theta \text{.............} \quad (1)$$

Where D: crysallite size, K=0.9, (βD: Full Width at Half Maximum FWHM), (0.154059 nm): The wavelength of an X-ray and the θ angle of diffraction.

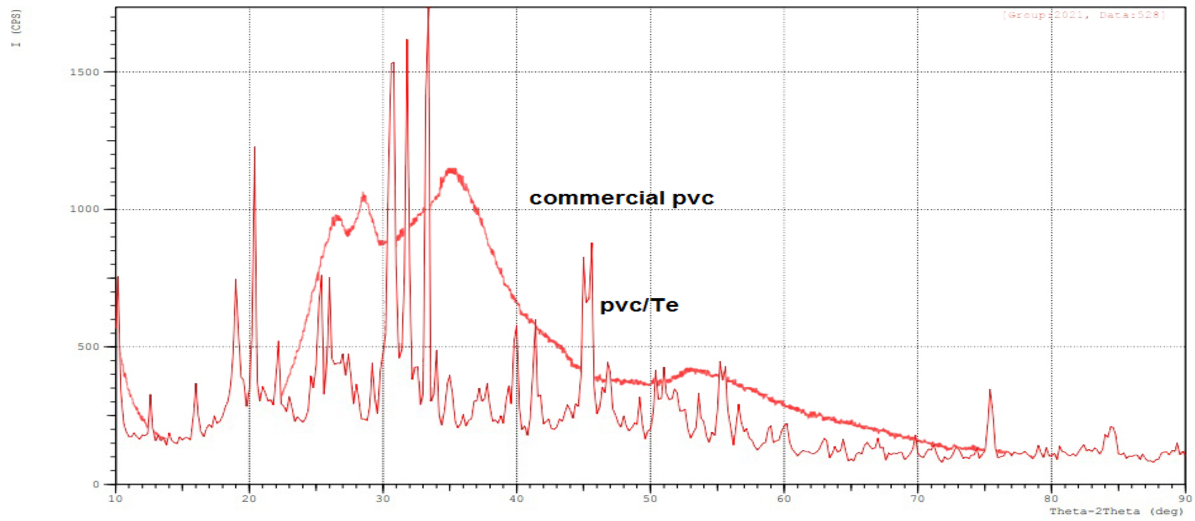

Figure 2 The XRD pattern of pure PVC and Polyvinyl Chloride doped Tellurium: the synthesized PVC/Te films have a cubic structure whereas pure PVC films have a low degree of crystallinity and a broad diffraction peak at low angles.

3. Element Analysis with EDX

Figure 3 shows Energy Dispersive X-ray (EDX) for PVC/ Te percentages of the elemental analysis which shows the weight percentage of polymer doped in the element tellurium 16%. This technique supplements the scanning electron microscope technology and investigates tellurium particle dispersion and interfacial compatibility in a PVC matrix.

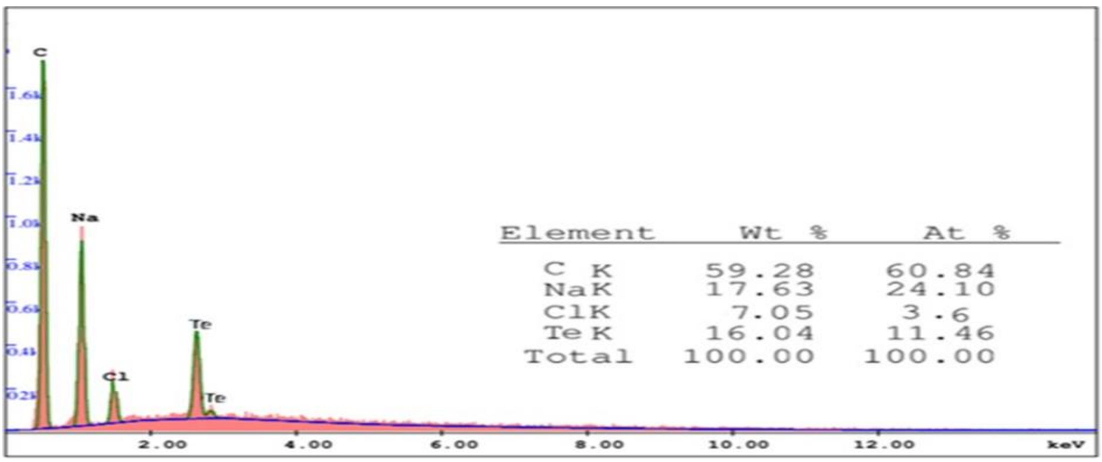

Figure 3. The elemental composition analysis by using EDX: carbon has the highest appearance percentage and the lowest atomic number among the other constituent elements.

4. Topography studies with FE-SEM

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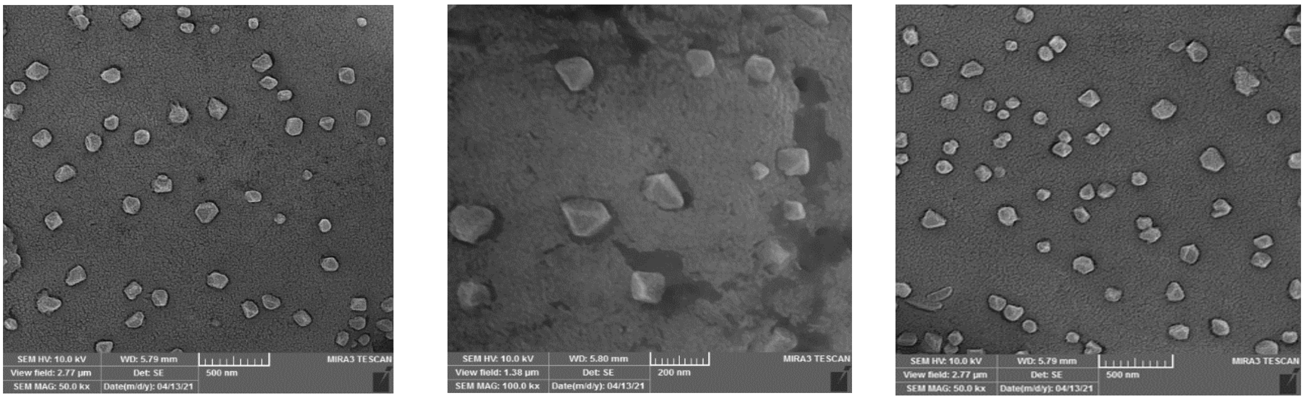
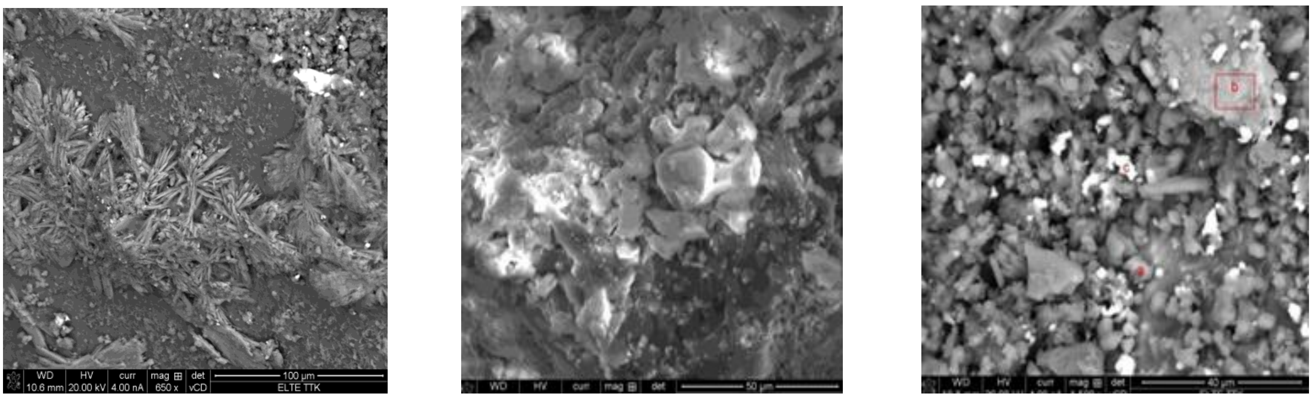
Figure 4 (a, b, c), FESEM shows the morphology of PVC/Te sample and the effect of surface modification at the level of morphology and aggregation. In this figure, three behaviors of PVC grafted Te can be shown. For example, figures a, b, and c are related to its subject for different electron beam energy where the sample exposed to the electronic energy that equals 10 KeV shows regular hexagonal geometries and is thought to represent tellurium crystals in PVC, as shown in Figure 4 (a).



(1) (2) (3)

Figure 4. a: The FESEM images of the morphology for PVC/Te composites with an electron beam of 10 keV applied, where tellurium molecules appear on the surface of the polymer in the form of crystals with regular faces (Hexagonal crystals).

However, when raising the electronic energy band value to the limit of 20KeV, it is noticed that the crystals begin to gather together and lose the hexagonal shape as shown in Figure (B), where the white areas are thought to belong to the tellurium element on the surface of the polymer that appears in the black space. Moreover, in Fig. 4 (c), 30KeV was applied to the sample; brighter white clusters occurred in the shape of the studied sample, which were believed to happen as a result of the breakage of the polymeric chains due to the high energy.



(1) (2) (3)

Figure 4. b The FESEM images of the morphology of PVC/Te compounds when an electron beam is increased to 20 kV, as the molecules shrink and the crystal regularity decreases.

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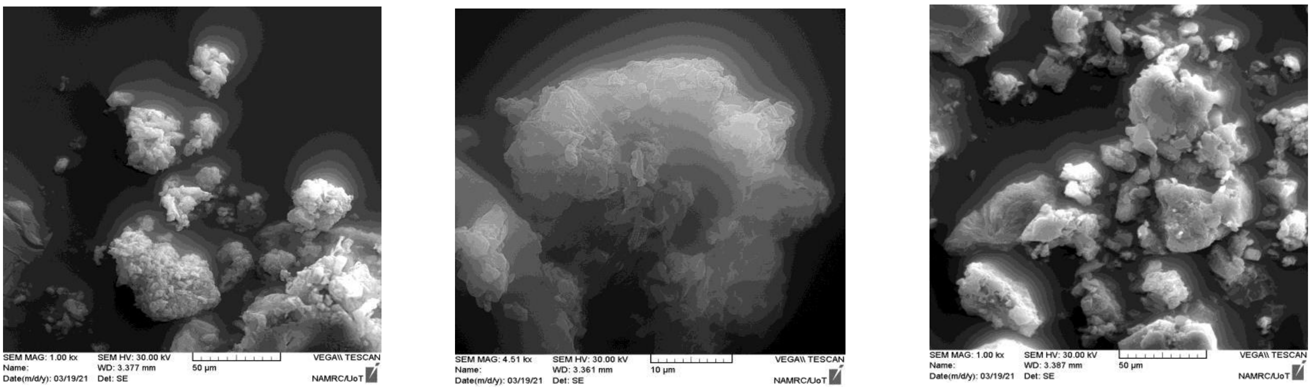

(1) (2) (3)

Figure 4. c The FESEM images of cleared deformation for PVC/Te composites with an electron beam of 30keV: the white brighter assembly composite particles are shown as an increased electron beam due to the breakdown polymer chain.

5. Thermogravimetric studies

TGA is a helpful tool for studying thermal decomposition kinetics, describing how the polymer degrades, determining the melting point and glass transition temperature, and studying the thermodynamics of thermal stability [27-30]. In this study, another evidence for enhancing polyvinyl chloride (PVC) polymer properties reached through the reaction of the above polymer with disodium ditelluride as an intermediate substance formed by the reaction of Tellurium powder with sodium borohydride in an alkaline solution. The study clearly showed that the polymer undergoes the exact expected mechanism of degradation (see Figure 4). Still, in the presence of Tellurium, Tellurium acts as a filler with a secondary interaction as shown in the microscopic study, where the PVC/Te compound showed a slow decomposition step while the stability was broached to 600oC in comparison with PVC alone, which decomposed at about 400oC. See Figure (5).

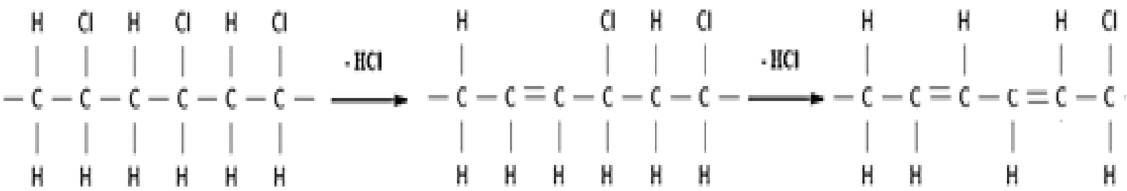

Figure 5. The schematic for expected mechanical Therma analysis of the PVC compound under static conditions achieve by losing the hydrochloric molecule in each stage of the thermal dissociation.

The amount of weight change of a substance as a function of temperature or time in an inert atmosphere is determined by the Thermogravimetric analysis.

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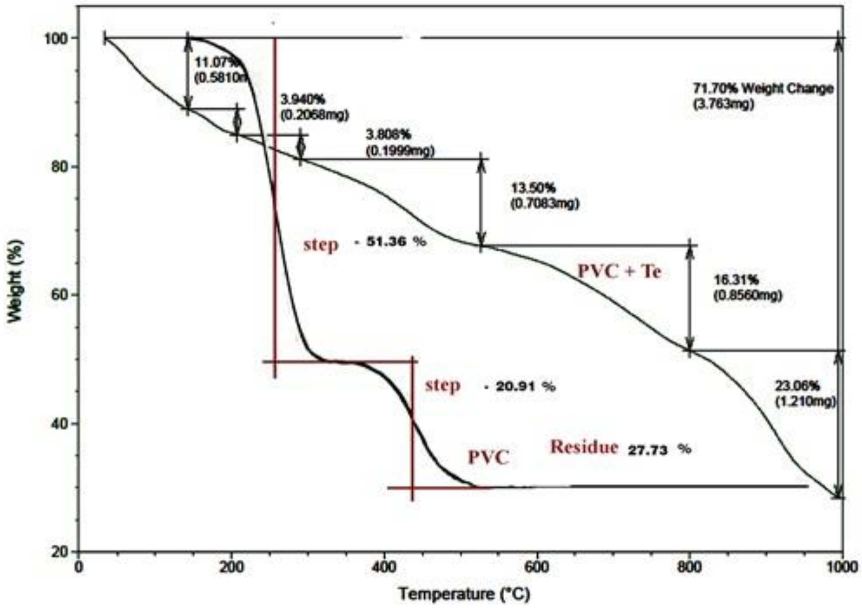

Figure 6. The Thermogravimetric TGA analysis of PVC and PVC doped Te

Conclusions

Under an inert atmosphere and in an alkaline medium, it is possible to modify the polyvinyl chloride PVC polymer to replace the labile chlorine atom with another atom, such as tellurium. By making use of certain techniques for the characterization of well-known chemical compounds such as FTIR and XRD, it is also possible to study changes that occur on the surface of the polymer by making use of the scanning electron microscope (SEM) and calculating the percentages of the elements using the EDX technique as a supplement to the SEM. Moreover, TGA-DTA can be used to study the thermal behaviour of the samples. It has been concluded that using the electron beam with different power will give other images for surface morphology. In contrast, in this work, we applied an electron beam with three energies (10 eV, 20 eV,30eV) and noted the atom's regular shape loss with the increased power of the electron beam in the scanning electron beam; we believed this occurs because breaking polymer chains where the molecules clump among themselves and thus influence the crystal structure of the compound. The technician advises the examiner not to shed high on electronic energy when examining molecules containing a polymer part in their composition in SEM. According to the XDR pattern, polyvinyl chloride doped Tellurium Compound has high crystallinity, whereas pure PVC is amorphous; the newly prepared compound demonstrated more excellent thermal stability than PVC alone.

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