

## Research Article

# Investigation of the Effects of Titanate as Coupling Agent and Some Inorganic Nanoparticles as Fillers on Mechanical Properties and Morphology of Soft PVC

Morteza Hajian, Gholam Ali Koohmareh, and Afsaneh Mostaghazi

*Department of Chemistry, College of Science, University of Isfahan, Isfahan 81746-73441, Iran*

Correspondence should be addressed to Morteza Hajian, m.hajian@chem.ui.ac.ir

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The effects of titanate as a coupling agent and some particulate nanoscale particles such as  $\text{TiO}_2$ ,  $\text{CaCO}_3$ , and  $\text{ZnO}$  on thermal and mechanical properties of emulsion polyvinylchloride (E-PVC) were investigated by thermogravimetric analysis (TGA), and mechanical tests. In this research, it was found that, in the presence of nanoparticles of  $\text{CaCO}_3$ ,  $\text{TiO}_2$ , and  $\text{ZnO}$ , the peak temperature of dehydrochlorination of E-PVC was shifted to higher temperatures, and the rate of mass loss was decreased. Also results of differential scanning calorimetry showed that the addition of nanoparticle of  $\text{CaCO}_3$ ,  $\text{TiO}_2$ , and  $\text{ZnO}$  led to an increase in glass transition temperature. The impact strength, elastic modulus and toughness of the samples were enhanced after addition of 0–10 part of filler in hundred parts of resin (phr) nano- $\text{CaCO}_3$ , nano- $\text{TiO}_2$ , and nano- $\text{ZnO}$  due to improvement of compatibility of the polymer and the nano-particles. Also UV and thermal stability of the samples were enhanced by means of the nanoparticles. It was found that, in the presence of titanate as coupling agent, content of additives that could be used in the composite of PVC shifts to higher amounts.

## 1. Introduction

Generally, PVC as a commodity plastic has been widely used in industrial fields such as outdoor applications, house siding panels, wastewater tubes, window profiles, and synthetic leather for many years, due to its good properties, such as nonflammability, low-cost, and formulating versatility. However, its low toughness and heat-softening temperature limit its application for some purposes [1, 2]. PVC is degraded thermally during processing and photochemically during applications with solar irradiation, UV light, and also by oxygen of atmosphere via radical mechanism. In contrast to some other polymeric materials, moisture is not an important factor to deteriorate its properties. Oxidation reactions are investigated by the growth of the IR absorption bonds for C=O around 1710 to 1720  $\text{cm}^{-1}$  [3, 4].

In recent years, nanocomposites based on PVC attracted much attention. Developing nanocomposites based upon

polymers and nanoscale fillers has been an attractive approach to achieving good properties. Various nanoscale fillers, including silica, mica, calcium carbonates, titanium oxide, and some other nanoparticles have been reported to enhance mechanical and thermal properties of polymers, such as toughness, stiffness, impact strength, elongation percent, and modulus [5–7].

Nano- $\text{CaCO}_3$  is one of the most common spherical nanoscale fillers used in preparation of nano composite suspension PVC. A study on nano- $\text{CaCO}_3$ -polypropylene (PP) composites revealed the dramatic toughening effect of nano- $\text{CaCO}_3$ , but because of the nucleating effect of nano- $\text{CaCO}_3$  the yield strength of PP was slightly decreased [8, 9]. Nano- $\text{TiO}_2$  and nano- $\text{ZnO}$  are common pigments that are used as fillers in preparation of nanocomposites. The roles of  $\text{TiO}_2$  and  $\text{ZnO}$  as pigments in the degradation process have not been completely elucidated, and conclusions are contradictory. For many researchers there is a question: do  $\text{TiO}_2$  and

TABLE 1: Sample contents.

Sample number	Sample code	PVC (gr)	DOP (gr)	CaCO <sub>3</sub> (gr)	Nano-CaCO <sub>3</sub> (gr)	TiO <sub>2</sub> (gr)	Nano-TiO <sub>2</sub> (gr)	ZnO (gr)	Nano-ZnO (gr)	Titanate (gr)
1	Control	10	5	0	0	0	0	0	0	0
2	C <sub>1</sub>	10	5	0.2	0	0	0	0	0	0
3	C <sub>2</sub>	10	5	0.4	0	0	0	0	0	0
4	C <sub>3</sub>	10	5	0.6	0	0	0	0	0	0
5	C <sub>4</sub>	10	5	0.8	0	0	0	0	0	0
6	C <sub>5</sub>	10	5	1.0	0	0	0	0	0	0
7	C <sub>6</sub>	10	5	1.2	0	0	0	0	0	0
8	nC1	10	5	0	0.2	0	0	0	0	0
9	nC2	10	5	0	0.4	0	0	0	0	0
10	nC3	10	5	0	0.6	0	0	0	0	0
11	nC4	10	5	0	0.8	0	0	0	0	0
12	nC5	10	5	0	1.0	0	0	0	0	0
13	nC6	10	5	0	1.2	0	0	0	0	0
14	nC7	10	5	0	1.4	0	0	0	0	0
15	nC8	10	5	0	1.0	0	0	0	0	1.0
16	nC9	10	5	0	2.0	0	0	0	0	2.0
17	nC10	10	5	0	3.0	0	0	0	0	3.0
18	nC11	10	5	0	4.0	0	0	0	0	4.0
19	nC12	10	5	0	5.0	0	0	0	0	5.0
20	nC13	10	5	0	6.0	0	0	0	0	6.0
21	T <sub>1</sub>	10	5	0	0	0.2	0	0	0	0
22	T <sub>2</sub>	10	5	0	0	0.4	0	0	0	0
23	T <sub>3</sub>	10	5	0	0	0.6	0	0	0	0
24	T <sub>4</sub>	10	5	0	0	0.8	0	0	0	0
25	T <sub>5</sub>	10	5	0	0	1.0	0	0	0	0
26	T <sub>6</sub>	10	5	0	0	1.2	0	0	0	0
27	nT <sub>1</sub>	10	5	0	0	0	0.2	0	0	0
28	nT <sub>2</sub>	10	5	0	0	0	0.4	0	0	0
29	nT <sub>3</sub>	10	5	0	0	0	0.6	0	0	0
30	nT <sub>4</sub>	10	5	0	0	0	0.8	0	0	0
31	nT <sub>5</sub>	10	5	0	0	0	1.0	0	0	0
32	nT <sub>6</sub>	10	5	0	0	0	1.2	0	0	0
33	nT <sub>7</sub>	10	5	0	0	0	1.0	0	0	1.0
34	nT <sub>8</sub>	10	5	0	0	0	2.0	0	0	2.0
35	Z <sub>1</sub>	10	5	0	0	0	0	0.2	0	0
36	Z <sub>2</sub>	10	5	0	0	0	0	0.4	0	0
37	Z <sub>3</sub>	10	5	0	0	0	0	0.6	0	0
38	Z <sub>4</sub>	10	5	0	0	0	0	0.8	0	0
39	Z <sub>5</sub>	10	5	0	0	0	0	1.0	0	0
40	Z <sub>6</sub>	10	5	0	0	0	0	1.2	0	0
41	nZ <sub>1</sub>	10	5	0	0	0	0	0	0.2	0
42	nZ <sub>2</sub>	10	5	0	0	0	0	0	0.4	0
43	nZ <sub>3</sub>	10	5	0	0	0	0	0	0.6	0
44	nZ <sub>4</sub>	10	5	0	0	0	0	0	0.8	0
45	nZ <sub>5</sub>	10	5	0	0	0	0	0	1.0	0
46	nZ <sub>6</sub>	10	5	0	0	0	0	0	1.2	0
47	nZ <sub>7</sub>	10	5	0	0	0	0	0	1.0	1.0
48	nZ <sub>8</sub>	10	5	0	0	0	0	0	2.0	2.0

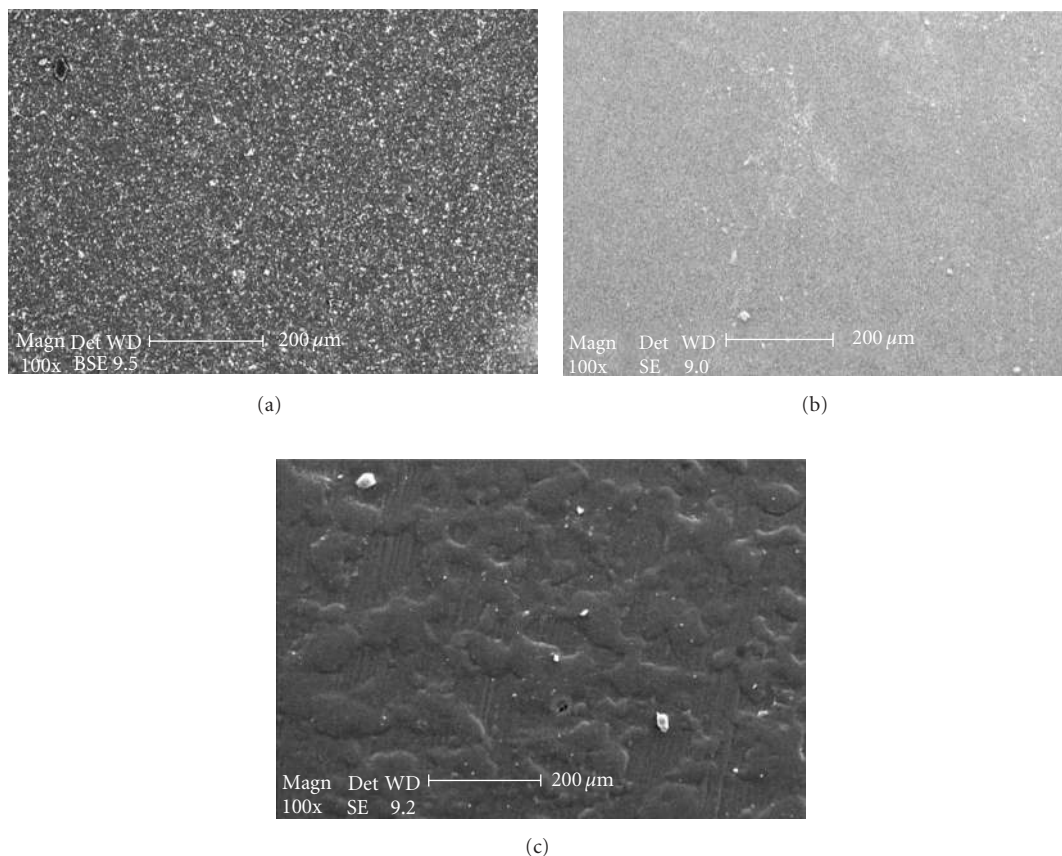


FIGURE 1: (a) SEM micrograph of sample code, C<sub>5</sub>. (b) SEM micrograph of sample code, nC<sub>5</sub>. (c) SEM micrograph of sample code, nC<sub>8</sub>.

ZnO act predominantly as physical UV absorbers and protect PVC, or do they act as photocatalyst for degradation of PVC?

The investigations of polymer scientists support the first view while application-technological experience is mixed.

To improve the overall quality of composites and treat the surface of fillers, coupling agents were used. The improvements are more apparent when composites are exposed to high humidity and high temperature. Coupling agents enhance adhesion between the filler and the polymer. They create strong bonds between their surfaces. Efficient coupling agents are silanes, and organotitanates, which are easily dispersed and present a high chemical resistance.

Organo titanate coupling agents create molecular bridges at the interface between inorganic fillers and polymer matrix. They overcome many of the limitations of incompatibility of polymers and inorganic fillers. The mechanism by which they couple different inorganic surfaces, which means that they are suitable not only for fillers having surface hydroxyl groups, but also for carbonates, carbon black, and other fillers that do not respond to silanes [10]. They have the advantages of forming only a monomolecular layer. The absence of a multimolecular layer at the interface and the chemical structure of titanates, modify the surface energy of filler particles in such a way that the melt viscosities of polymers are lower than the other type of coupling agent [11]. So the use of titanates is preferred. It has also been researched that the addition of titanates to nanocomposites,

improved the mechanical properties of polymer [5, 11–13]. Addition of montmorillonite in polyethylene (PE) and poly(ethylene terephthalate) (PET) in a twin-screw extruder caused a consideration improvement in compatibility and tensile strength of the composite [14, 15]. In this paper the effects of titanate as coupling agent and some nanoinorganic pigments and fillers on mechanical properties and morphology of the soft PVC composites were studied.

## 2. Experimental

**2.1. Materials and Sample Preparation.** Emulsion PVC ( $M_w = 35000$ ) was produced by European group of companies. Nano-CaCO<sub>3</sub> (NPCC 2-01, surface modifies with stearic acid) with particle size of 29 nm was produced by nanomaterial technology Pte. Chemical Co; Ltd; Singapore. Nano-TiO<sub>2</sub> (type HQ lab)-(P-38) with particle size of 80 nm was prepared by nano-photo-catalysts chemical company; China. Dioctyl trephthalate (DOP) was produced by Merck Company. Organo titanate coupling agent was industry grade product, ((2,2-bis(allyloxymethyl)butoxy) tris((bis(octyloxy)phosphoryl) methyl)(hydroxy) phosphoxy) titanium). Nano-CaCO<sub>3</sub>, nano-TiO<sub>2</sub>, and nano-ZnO were dried at 80°C for 24 h in a vacuum oven. PVC, DOP, and nanoparticles were mixed at a constant speed to give a homogenous PVC compound. ASTM D638 test method was used for tensile properties of plastics such as

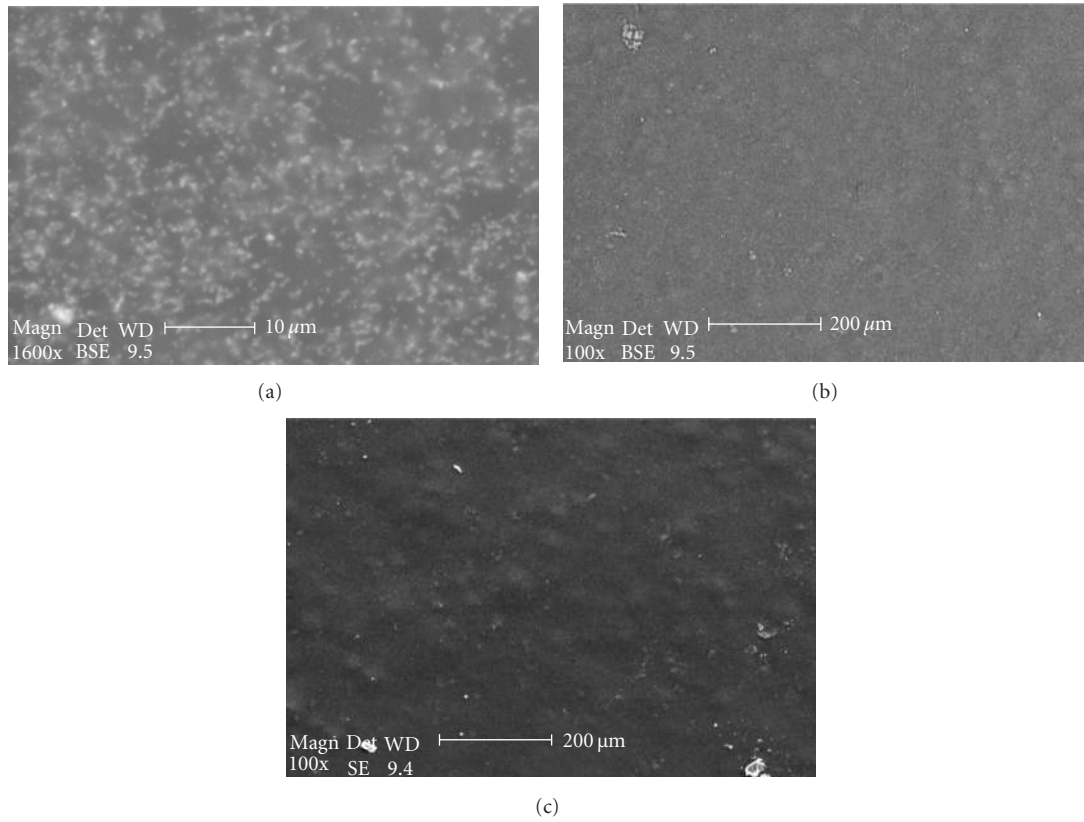


FIGURE 2: (a) SEM micrograph of sample code, T<sub>5</sub>. (b) SEM micrograph of sample code, nT<sub>5</sub>. (c) SEM micrograph of sample code, nT<sub>7</sub>.

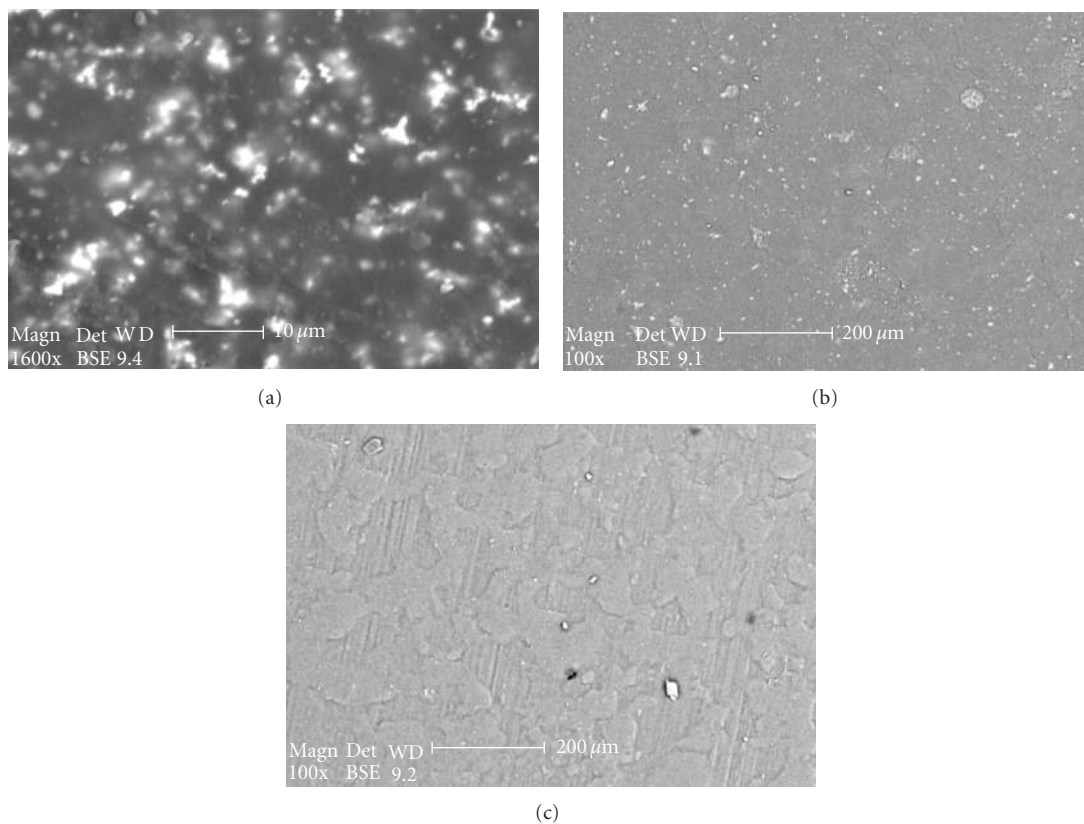


FIGURE 3: (a) SEM micrograph of sample code, Z<sub>5</sub>. (b) SEM micrograph of sample code, nZ<sub>5</sub>. (c) SEM micrograph of sample code, nZ<sub>7</sub>.

TABLE 2: Mechanical properties of prepared composites.

Formulation: PVC resin ( $M_W = 35000$ )—Plasticizer (DOP)—Filler ( $\text{CaCO}_3$ )				
Property	Modulus, psi	% Elongation at break	Tensile strength, psi	Energy, J
<i>Control</i>	6.3	92.2	5.8	1175
C <sub>1</sub>	8.9	88.9	7	1548
C <sub>2</sub>	9.8	86.5	7.6	1698
C <sub>3</sub>	11.3	82	9.26	2003
C <sub>4</sub>	12.7	79	10.3	2240
C <sub>5</sub>	14.4	76.5	11	2457
C <sub>6</sub>	14.8	63	9.32	1590
Formulation: PVC resin ( $M_W = 35000$ )—Plasticizer (DOP)—Filler ( $\text{TiO}_2$ )				
Property	Modulus, psi	% Elongation at break	Tensile strength, psi	Energy, J
<i>Control</i>	6.3	92.2	5.8	1175
T <sub>1</sub>	7.25	87.9	6.37	1448
T <sub>2</sub>	7.67	81.2	6.22	1450
T <sub>3</sub>	10.4	78.01	8.1	1820
T <sub>4</sub>	11.2	74.86	8.37	1853
T <sub>5</sub>	11.4	70.8	8.12	1515
T <sub>6</sub>	12.38	70.4	8.71	1435
Formulation: PVC resin ( $M_W = 35000$ )—Plasticizer (DOP)—Filler (ZnO)				
Property	Modulus, psi	% Elongation at break	Tensile strength, psi	Energy, J
<i>Control</i>	6.3	92.2	5.8	1175
Z <sub>1</sub>	9.6	69.9	6.71	1032
Z <sub>2</sub>	10.7	65.4	7.05	1029
Z <sub>3</sub>	11.37	65.07	7.39	1035
Z <sub>4</sub>	13.27	62.6	8.31	1436
Z <sub>5</sub>	13.31	62.4	8.31	1427
Z <sub>6</sub>	13.86	43.16	5.98	528

PVC. Specimen dimension for thickness showed in this test method purposed type IV for specimen.

**2.1.1. Synthesis of Nano-ZnO Particles.** About 20 gr of zinc sulfate dehydrate was dissolved in minimum amount of distilled water, and the solution volume increased to 50 mL. Then NaOH solution (4 molar) was added by dropping funnel until a white precipitate was prepared which was then converted to a colloidal solution. Addition of NaOH was continued to pH 7. The solution was then mixed for 12 h. The precipitate was filtered and washed with distilled water several times. The resulted solid was dried in oven at 100°C for 3 h and finally was heated at 400°C for 2 h to afford nano-ZnO particles.

**2.1.2. Sample Preparation.** In a 200 mL baker, PVC (10 gr), DOP (5 gr), and different amounts of fillers according to Table 1 were mixed completely for 5 minutes by using mechanical stirrer to gave a homogeneous paste.

**2.1.3. Film Preparation.** The homogeneous paste was molded on an aluminum sheet for 0.3 mm in thickness and cured at 180°C for 10 min in a vacuum oven, followed by cooling to room temperature [8].

## 2.2. Characterization

**2.2.1. Methods and Instrument.** Surfaces of composites for determining dispersion of nanoparticle in PVC matrix were observed by scanning electron microscopy (SEM) with a JEM 1600 EX apparatus running at an acceleration voltage of 80 kV.

X-Ray diffraction was performed to determine the diameters of nanoparticles by X-Ray Diffractometer, Bruker, D8ADVANCE.

Tensile tests were performed at room temperature at a crosshead speed of 50 mm/min using an Instron tensile tester from Santam Co. (SMT-5); BONSHIN, Model: DBBP-500 according to ASTM D 638M. The test specimen shall conform to the dimensions showed in this standard test method. Type IV specimen was used for testing nonrigid plastics with a thickness of 4 mm or less. Thermogravimetric analysis was performed by Metler TG analyzer (TG-50) to determine the thermal stability of polymer composites.

## 3. Results and Discussion

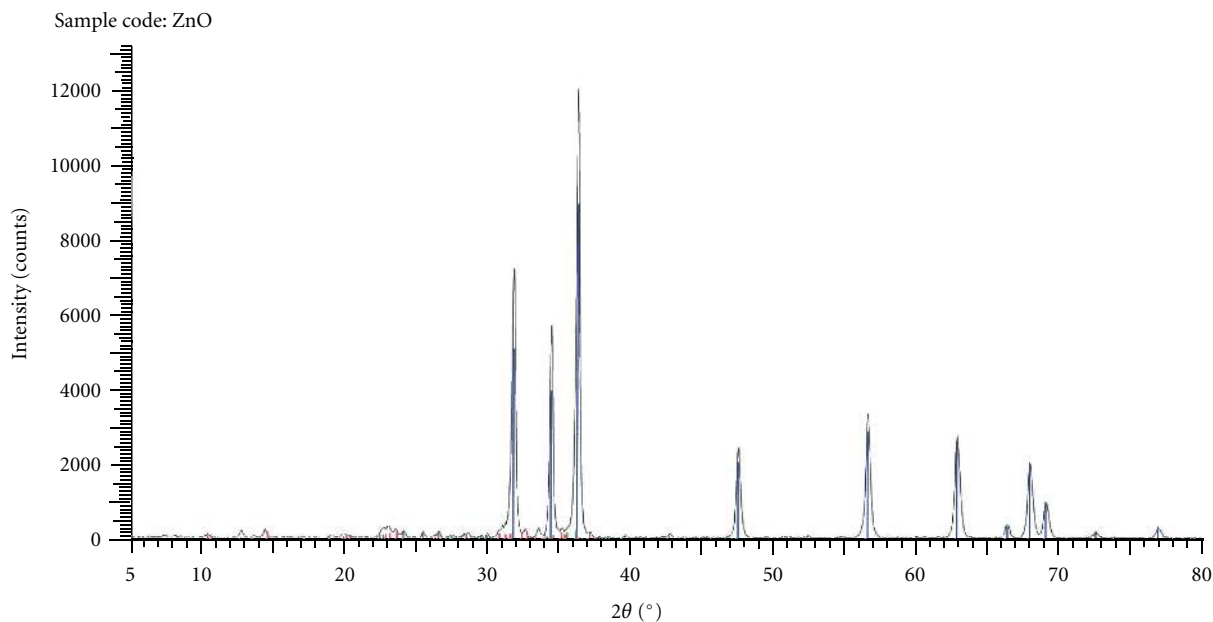
**3.1. Morphology Observation.** To investigate the effects of  $\text{CaCO}_3$ ,  $\text{TiO}_2$  and ZnO, and also their nanoparticles on mechanical properties and morphology of PVC, different

TABLE 3: Mechanical properties of prepared nanocomposites.

Formulation: PVC resin ( $M_W = 35000$ )—Plasticizer (DOP)—Filler (nano- $\text{CaCO}_3$ )				
Property	Modulus, psi	% Elongation at break	Tensile strength, psi	Energy, J
<i>Control</i>	6.3	92.2	5.8	1175
nC1	7.7	89.9	6.92	1496
nC2	9.4	84	7.91	1680
nC3	11.4	82.2	9.4	2100
nC4	12.1	81	9.8	2245
nC5	13.2	78	10.29	2477
nC6	13.2	80	10.6	2490
nC7	13.5	72	9.72	1840
Formulation: PVC resin ( $M_W = 35000$ )—Plasticizer (DOP)—Filler (nano- $\text{TiO}_2$ )				
Property	Modulus, psi	% Elongation at break	Tensile strength, psi	Energy, J
<i>Control</i>	6.3	92.2	5.8	1175
nT <sub>1</sub>	7.18	88.2	6.33	1490
nT <sub>2</sub>	8.7	82	7.13	1580
nT <sub>3</sub>	10.8	78.8	8.51	1895
nT <sub>4</sub>	11.3	76.2	8.06	1899
nT <sub>5</sub>	11.42	73.35	8.37	1607
nT <sub>6</sub>	12.04	69.9	8.41	1538
Formulation: PVC resin ( $M_W = 35000$ )—Plasticizer (DOP)—Filler (nano- $\text{ZnO}$ )				
Property	Modulus, psi	% Elongation at break	Tensile strength, psi	Energy, J
<i>Control</i>	6.3	92.2	5.8	1175
nZ <sub>1</sub>	9.55	72.3	6.9	1108
nZ <sub>2</sub>	10.23	70.55	7.21	1125
nZ <sub>3</sub>	11.2	68.8	7.7	1254
nZ <sub>4</sub>	13.3	64.2	8.53	1670
nZ <sub>5</sub>	13.35	62.5	8.57	1554
nZ <sub>6</sub>	13.65	57.8	7.88	1002

TABLE 4: Mechanical properties of prepared nanocomposites in the presence of titanate coupling agent.

Formulation: PVC resin ( $M_W = 35000$ )—Plasticizer (DOP)—Filler (nano- $\text{CaCO}_3$ )—Titanate (% by weight of nano- $\text{CaCO}_3$ )				
Property	Modulus, psi	% Elongation at break	Tensile strength, psi	Energy, J
<i>Control</i>	14.4	76.5	11	2457
nC8	12.72	110.2	14	3338
nC9	12.96	74.35	9.62	1812
nC10	12.89	64.6	8.2	1436
nC11	14.9	45.5	6.8	730.3
nC12	14.4	42	6.04	495.5
nC13	15.1	40.3	6.08	463.09
Formulation: PVC resin ( $M_W = 35000$ )—Plasticizer (DOP)—Filler (nano- $\text{TiO}_2$ )—Titanate (% by weight of nano- $\text{TiO}_2$ )				
Property	Modulus, psi	% Elongation at break	Tensile strength, psi	Energy, J
<i>Control</i>	11.4	70.8	8.12	1515
nT <sub>7</sub>	9.9	107.5	10.64	2995
nT <sub>8</sub>	11.8	62	7.31	1022.2
Formulation: PVC resin ( $M_W = 35000$ )—Plasticizer (DOP)—Filler (nano- $\text{ZnO}$ )—Titanate (% by weight of nano- $\text{ZnO}$ )				
Property	Modulus, psi	% Elongation at break	Tensile strength, psi	Energy, J
<i>Control</i>	13.31	62.4	8.31	1427
nZ <sub>7</sub>	11.9	89.5	10.64	2498.5
nZ <sub>8</sub>	13.8	62.2	8.58	1118



Sample identification and quantification




Line color	Compound name	Formula	PDF number	Concentration (%W/W)
	Zincite, syn	ZnO	36-1451	95.2
	Sodium zinc sulfate	$\gamma$ -Na <sub>2</sub> Zn(SO <sub>4</sub> ) <sub>2</sub>	24-1171	2.2
	Zinc oxide sulfate	Zn <sub>3</sub> O(SO <sub>4</sub> ) <sub>2</sub>	32-1475	2.6

FIGURE 4: The XRD micrograph of synthesized ZnO nanoparticles.

samples containing different amounts of the fillers were prepared (Table 1). The surface morphology of the prepared samples were investigated by Scanning Electron Microscopy (SEM), and the results were shown in Figures 1, 2, and 3. As shown in these figures, dispersion of nanoparticle is better in PVC films.

It is also observed that in the presence of titanate as coupling agent, a nanoparticle homogeneously dispersed in PVC matrix indicating good compatibility of nanofiller and polymer.

The XRD micrograph of synthesized ZnO nanoparticles shows the nanometric size of particles in Figure 4. It was obtained from this micrograph (by using Scherrer equation) that the size of nanoparticles of ZnO used in PVC is 21 nanometer.

**3.2. Tensile Properties.** Mechanical properties of the prepared composites and nanocomposites were investigated, and the results were shown in Tables 2, 3, and 4. It could be obtained from the tables that the elongation at break of both composites and nanocomposites for all fillers was decreased with increase of the filler contents, but it can be seen that the rate of loss is more in composite than in the related nano composite. It has also been obtained from the tables that elastic modulus of the nanocomposites could be increased with increase of the nanoparticle content. But the value of modulus in nanocomposites is lower in contrast with PVC/CaCO<sub>3</sub> composites. It can be concluded from this result

TABLE 5: Thermal analysis data of composites obtained by TG/DTG.

Sample code	First Peak 1 (°C)	Second Peak 1 (°C)	Third Peak 1 (°C)	δm % (Remained polymer)
Reference	244.3	291	445	13.45%
nC <sub>5</sub>	—	302.7	447.3	15.30%
nT <sub>5</sub>	—	300	447.3	15.39%
nZ <sub>5</sub>	—	303	445	26.30%

that, beside the inherent properties of polymer matrix, the dispersion state of the fillers also affects on mechanical properties such as elastic modulus of composites.

Table 4 shows mechanical properties of the nanocomposites in the presence of titanate as coupling agent. It can be obtained that addition of titanate to PVC compound led to an increase in elongation percent. Also the content of filler that could be used increased to 40 phr for nano-CaCO<sub>3</sub> and 20 phr for nano-TiO<sub>2</sub> and nano-ZnO.

**3.3. Thermal Analysis.** The thermal stability of the composites was investigated by TG and DTG. All the composites had a weight loss peak near 290°C because of loss of dioctyl phthalate (DOP) and HCl. The DTG of the PVC film without inorganic fillers (reference sample) was shown in Figure 5. A first small peak at 244°C shown in this figure is related to loss of HCl, the second at 291°C for loss of DOP and the third

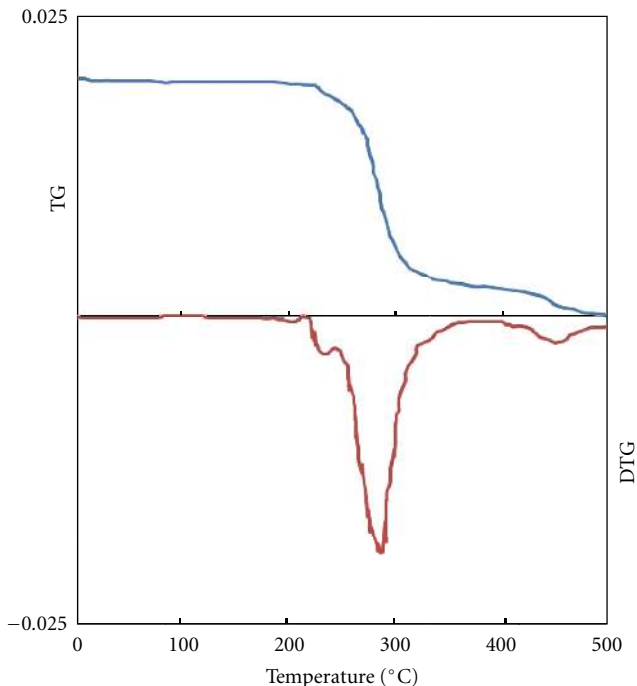


FIGURE 5: TG/DTG thermogram of reference sample.

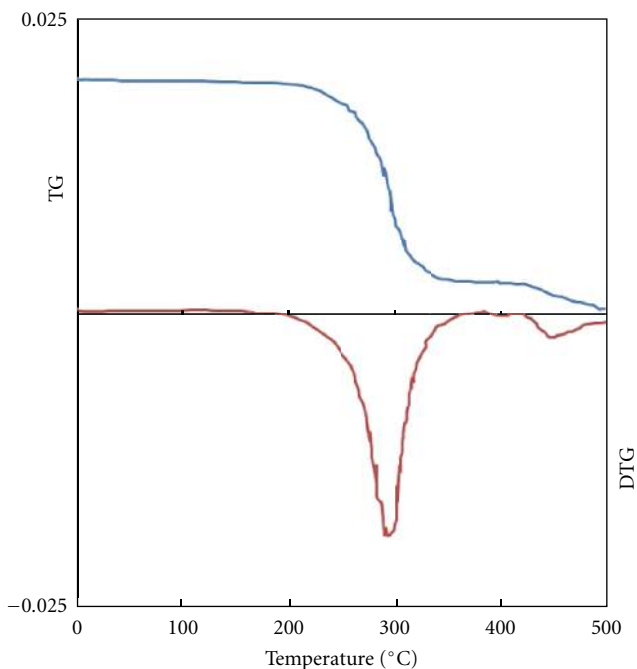


FIGURE 6: TG/DTG thermogram of sample  $nC_5$ .

peak at 445°C for final degradation. For the other samples, the first weight loss related to loss of HCl near 244°C in reference sample was disappeared, and also the temperature of loss of DOP was increased to near 300°C which confirmed thermal stability of these samples.

A thermal analysis curve related to PVC/nano- $\text{CaCO}_3$  (sample number  $nC_5$ ) was shown in Figure 6.

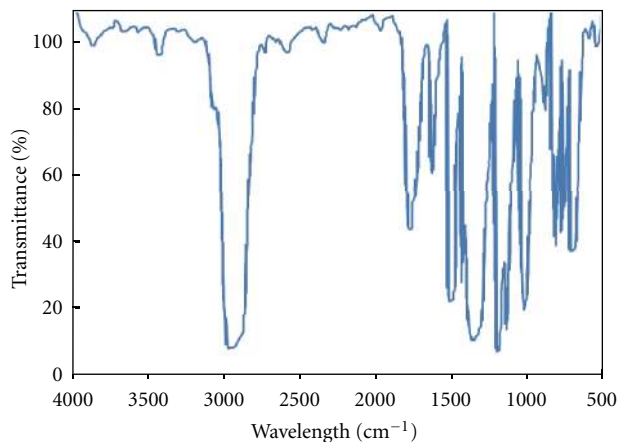


FIGURE 7: FTIR spectrum of reference sample.

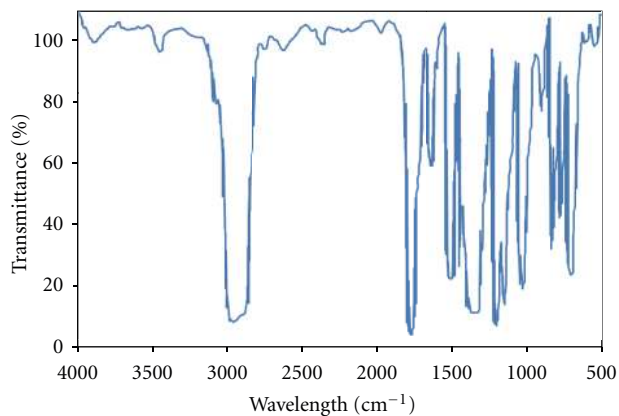


FIGURE 8: FTIR spectrum of reference sample after treating 45 days with UV light.

For some other samples, the thermal analysis data were recorded, and the results are collected in Table 5.

**3.4. UV Studies.** FTIR spectrum of the reference sample showed an absorption for carbonyl group which was related to DOP (Figure 7). In order to investigate the UV stabilization of the samples, first a reference sample was treated with UV light for 45 days. FTIR spectra of this sample after treating with UV light (Figure 8) showed an increase in intensity of carbonyl absorption which was related to PVC degradation with UV light. In other samples with nanofillers, the intensity of this peak decreased which was confirmed the UV stability of these fillers. As a typical example, FTIR spectrum of sample code ( $nZ_5$ ) after treating 45 days with UV light was shown in (Figure 9).

## 4. Conclusion

PVC composites and nanocomposites were prepared by PVC plastisol and mixing process at room temperature. Nanoparticles are well dispersed in the polymer matrix by titanate as a coupling agent. In this process addition of titanate acts as a compatibilizer and cause increases of tensile



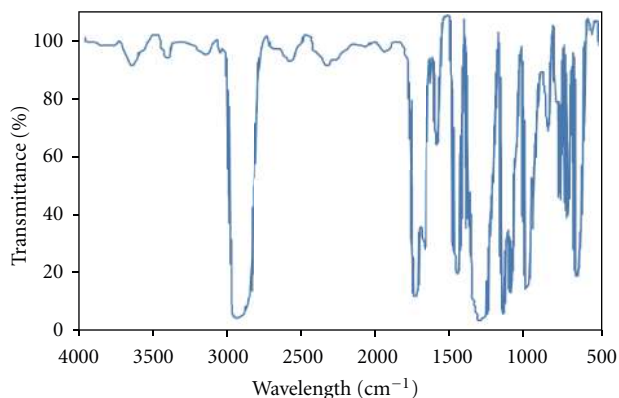


FIGURE 9: FTIR spectrum of sample code (nZ<sub>5</sub>) after treating 45 days with UV light.

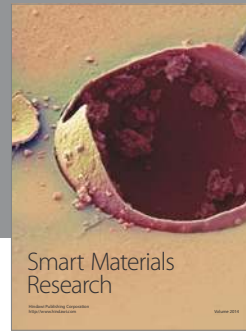
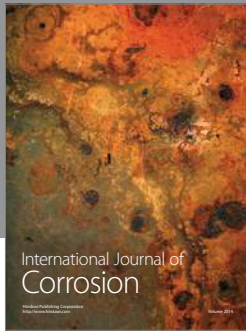
strength and modulus of the samples while the content of nanoparticle was less than 10 phr. The impact strength and elastic modulus of E-PVC could be increased by addition of nanoparticles, and higher toughness could be also achieved from these composites.

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