IMPACTS OF NANOSCALE INCLUSIONS ON FIRE RETARDANCY, THERMAL STABILITY, AND MECHANICAL PROPERTIES OF POLYMERIC PVC NANOCOMPOSITES

A. Ghazinezami, W. S. Khan, A. Jabbarnia, R. Asmatulu*

ABSTRACT

A number of different nanoscale inclusions including nanoclay, nanotalc, and graphene were incorporated with polyvinyl chloride (PVC), dispersed in N,N-Dimethylacetmide (DMAC) and cast in rectangular aluminum (Al) molds prior to the testing. The fire retardancy, thermal stability, and mechanical properties of the PVC nanocomposites were determined using the ASTM UL 94 standard, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and microtensile test units. Surface morphology studies of the resultant materials were also carried out using scanning electron microscopy (SEM). Test results showed that the fire retardancy, thermal stability, and mechanical properties of the PVC nanocomposites were significantly enhanced in the presence of nanoscale inclusions. Among the inclusions, graphene had the major impact on improving the physical properties, which may be because of its higher thermal conductivity, mechanical strength, size, and shape. Polymers have a wide range of applications in daily life, but they are highly flammable and mechanically not stable for different applications. This study has shown that the weakness of the PVC could be significantly enhanced by incorporating nanoscale inclusions for various industrial purposes.

Keywords: Nanoscale Inclusions, PVC Nanocomposites, Fire Retardancy, Physical Properties

INTRODUCTION

Polymers have a wide range of industrial applications and greatly enhance the quality of daily modern life because of their extraordinary properties, such as low weight, cost, flexibility, and manufacturability [1-3]. These materials have been replacing traditional inorganic materials, including natural polymers, metals, ceramics, composites, and wood. Since polymers are flammable, they are incorporated with flame-retardant additives in order to reduce their flammability. Nanoparticle-based filler materials are outstanding candidates for this purpose because they not only improve the physical and mechanical properties of polymers but also reduce polymer flammability. Previously, halogenated additives were very common, but due to their suspected adverse effects on the environment, they are banned in many countries [2-6]. Currently, the flammability of polymers and discovering proper environment-friendly fire-retardant polymers are major challenges for researchers.

Nanofillers such as carbon nanotubes, graphene, nanotalc and clay are attractive materials, because addition of small amount of nanofillers can improve mechanical, thermal and electrical properties of polymers without changing processability [28]. Nanofiller-based polymers show high flame-retardant efficiencies. Adding only a small amount (<5%) of nanofillers can reduce the peak heat release rate of polymer and thereby reduce the speed at which flame spread [4-8,28]. Polymers are being used in applications where specific mechanical, thermal and electrical properties are required [29]. PVC and fluoropolymers are intrinsically flame-retardant polymers [29]. Nanomaterials have proved themselves to be highly effective fire retardants. Clays, talc and graphene in particular deliver safer environmental profiles compared to the alternatives, as the materials are completely natural to begin with. Fire retardant nanomaterials have become a major business for many industries and can be found anywhere in modern society. Almost all plastic or polymer products such as computer cables, electrical appliances, cars, materials in airplanes, trains and ships from fabric of seats to all kind of plastic structure need flame retardant characteristics. [29-32]

The technical and scientific literature contains some very efficient strategies to improve the fire resistance of polymers, depending on their chemical structure, nature, and decomposition; the level of safety; and performance of the final products [2]. Polymer nanocomposites form barriers between the fuel and air, reducing the rate of ignition, flammability, and burning process [3]. The rheological properties of molten polymer

nanocomposites, which are different from those of virgin polymers, will significantly affect heat transfer through the material. The influence of polymer nanocomposite formation on the yield of toxic products from fire is generally studied using the ISO 19700 steady-state tube furnace, and in the early stages of burning, carbon monoxide and organo-irritants usually evolve, but under under-ventilated conditions, toxic products are formed, thus jeopardizing human life [3-7].

Polyvinyl chloride (PVC), an amorphous polymer, does not ignite easily and has a high ignition temperature (455°C). It is chemically stable and exhibits only a small change in mechanical strength. This viscoelastic material has small creep deformation due to limited molecular motion at room temperature. In a PVC molecular structure, the chlorine atom is bound to every other carbon chain, which results in a high resistance to oxidation, thus maintaining its performance for a long period of time. The heat release during the burning of PVC polymer is much less compared to heat released during the burning of polypropylene (PP) and polyethylene (PE), thus greatly preventing the spread of fire to nearby materials. PVC also possesses an oil/chemical resistance during its service.

Although PVC has a wide range of industrial applications and improves the quality of life, burning PVC at certain high temperatures can result in corrosive and toxic gases and smoke for an environmental damages. The aim of this study was to add nanoscale inclusions (nanoclay, nanotalc, and graphene) into the PVC in order to fabricate a nanocomposite through solution casting and determine its fire retardancy, thermal stability, and mechanical properties for various high-temperature applications in transportation, packaging, electronics, energy, and defense industries.

EXPERIMENTAL PROCEDURES
Materials

PVC powder chloride was purchased from Sigma-Aldrich, while N,N-Dimethylacetmide was purchased from Fisher Scientific. Nanoclay (Cloisite 30B), in a white powder form with a typical dry particle size/diameter of less than 10 μm and amine functionalized, was provided by Southern Products, Inc. Nanotalc (hydrous magnesium silicate) was provided by Luzenac, Inc. Graphene (N006-010-P), in powder form with a thickness of 10 to 20 nm and diameter of less than 14 μm, was purchased from Angstrom Materials, Inc. These materials and chemicals were used without any further modification.

Methods
Manufacturing PVC Nanocomposites

PVC was used as the matrix material, while DMAC was used as the solvent. Different weight percentages of nanoclay, nanotalc, and graphene were first added to DMAC and sonicated for about two hours, and then known amounts of PVC powders were added to the solution prior to stirring on a hot plate at 60oC for 12 hours. Special care was taken to ensure a homogeneous blend of the mixture without any agglomeration [7]. The solution was then transferred into a specially designed rectangular aluminum (Al) mold. The mold was sprayed with a release agent in order to facilitate easy removal of the specimens. The solution/dispersion was allowed to solidify at room temperature, and then the specimen was removed from the mold. Figure 1 shows the Al mold and casting of the PVC nanocomposites.

ASTM UL 94 V Test

One of the most common flame tests to determine the flammability of plastic materials is the ASTM UL 94 standard test, which identifies the tendency of materials to either self-extinguish or spread the flame once a specimen is ignited [4,5]. In the present study, vertical UL 94 tests were carried out to determine the flame retardancy of the prepared specimens. The specimen size follows the standards for the flame-retardancy tests. During the application of the flame, it is very important to maintain a constant distance between the burner and specimen. Specimens should be 12.7 cm x 1.7 cm in size with a thickness of 1.5 mm. Typically, there are three major ratings (V0, V1, and V2) for the typical UL 94 vertical test. V0 is the best rating among all three ratings [6].

Bunsen Burner Flame

Flame is a region where combustion takes place to burn the prepared specimen. The color of the flame generally depends on the substance and temperature. Hydrocarbon (propane) flames are either yellow or blue. A
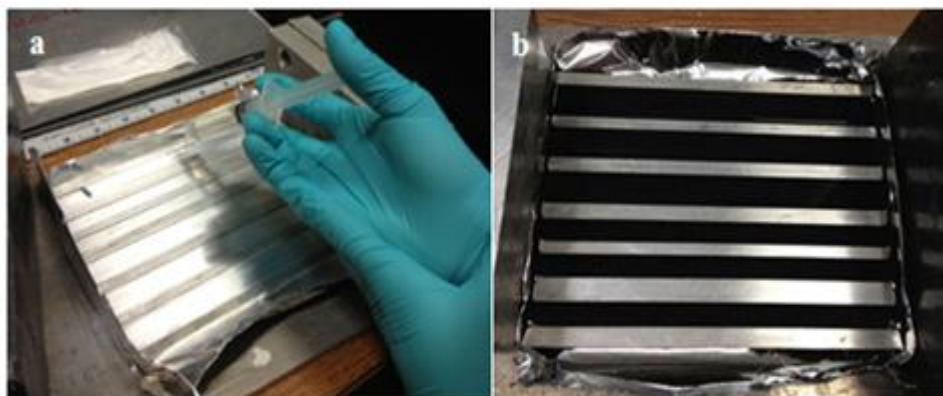
Figure 1. Casting pictures: (a) pure PVC, and (b) PVC incorporated with 20% graphene nanoflakes in Al molds

standard Bunsen burner with an inner diameter of 10 mm was chosen for the flammability tests. Specimens were vertically penetrated on the tip of the flame that had the highest temperature reading.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) is a thermal characterization test to examine the mass change of a specimen as a function of time and temperature in an isothermal mode. A TGA Q5000 (TA instrument) was employed for the prepared samples. In TGA, the mass change of the sample is measured in either relative percentage or absolute value in milligrams, and then test results are plotted to show the change in time or temperature. TGA is primarily influenced by an accurate heating rate and operating conditions. ML is the percentage loss of mass and is calculated from the ms (initial mass before heating) and mf (when temperature is reached to the end) using the following equation:

$$M_L = \frac{m_s - m_f}{m_s} \times 100 \qquad (1)$$

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is an analysis technique to measure the energy that is necessary to establish a nearly zero temperature difference between an inert reference material and a substance as it is subjected to identical conditions (temperature regime) in a controlled environment [7-9]. DSC determines several properties of polymers, such as glass transition temperature (Tg), melting point, degradation temperature, melting enthalpy, and crystallization. A DSC Q2000 (TA Instrument) interfaced with a personal computer (PC), was used to measure the thermal properties of the PVC nanocomposite samples. The Tg was investigated with a heating rate of 10oC/min and a nitrogen flow rate of 50 ml/min. The prepared samples were sealed in an aluminum pan (TA Instruments), and the measurements were conducted in the temperature range of 20oC to 350°C. A predetermined weight of each sample was used in this experiment. With the help of this analysis technique, very useful information can be extracted from the prepared specimen, including phase changes, crystallization, glass transition, and melting points.

Mechanical Properties

The microtensile test (PASCO Stress-strain Apparatus) was used to determine the mechanical properties of the prepared specimens. Both strain and stress values were measured by the compact stress-strain apparatus used in this experiment, which displayed both stress and strain during the process of specimen loading/stretching. The specimen was usually stretched by rotating the knob until the specimen broke up. The force required to stretch the sample was measured by a force sensor.

The other tests, including scanning electron microscopy (SEM), optical microscopy and visual observations were conducted throughout the tests, as well.

RESULTS AND DISCUSSION
ASTM UL 94 Tests

The vertical ASTM UL 94 test was employed to determine the flame retardancy of the prepared PVC nanocomposite specimens as is indicated in Table 1. All samples were weighed before and after the burning test. Based on the results, PVC with 0 wt.% of nanoparticles failed in the first ten-second burning period. The entire pure PVC sample was burned completely after the flame was started. From Table 1, specimens containing 10 wt.% nanoclay and graphene and also 20 wt.% of all nanoparticles showed the best fire-resistance properties compared to lower nanoscale concentrations. These are classified as V0, which is the best/highest rating against burning. Results confirmed that the fire-retardancy properties improved as the concentration of nanofiller materials increased in the PVC samples.

SEM analysis was performed on the PVC samples in order to determine changes in their surface morphological. Figure 2 shows SEM images of the PVC nanocomposites before and after burning tests. Figure 2a shows the surface of the burned PVC sample that failed after ten seconds. As can be seen in Figure 2b, nanoclay in the PVC sample was concentrated at the interface between the flame and the PVC matrix, forming a thick layer of nanoclay inorganic filler material. During the burning process, holes and bubbles were formed at the interface. These morphological changes are crucial in controlling the flame propagation since holes enclose the unburned inorganic nanomaterial layers.

Similar morphological changes were also observed after burning the samples having 20 wt.% nanotalc (Figure 2c). These morphological changes include swelling, bubbles, and hole formations. Holes and bubbles were concentrated at the interface between the PVC and air during the burning and drastically changed the flammability and fire-retardancy behaviors of the prepared PVC nanomaterials. Figure 2d shows morphological changes of the sample with 20 wt.% graphene after the burning process. As can be seen, bubbles are visible on the right side, indicating the presence of a thick layer of graphene on the surface. This confirms that graphene is an excellent conductor of heat and dissipates energy on the entire surface of the specimen prior to the burning process.

Thermogravimetric Analysis Tests

Thermogravimetric analysis quantifies the amount of moisture and volatile compounds present in the nanocomposites, weight loss, and thermal breakdown, and assists in determining the degradation mechanisms. TGA was used to characterize the decomposition and thermal stability of materials under various conditions and to determine the kinetics of the physicochemical processes that may take place during the decomposition process.

Table 1. UL 94 V test rating for all prepared PVC nanocomposite samples

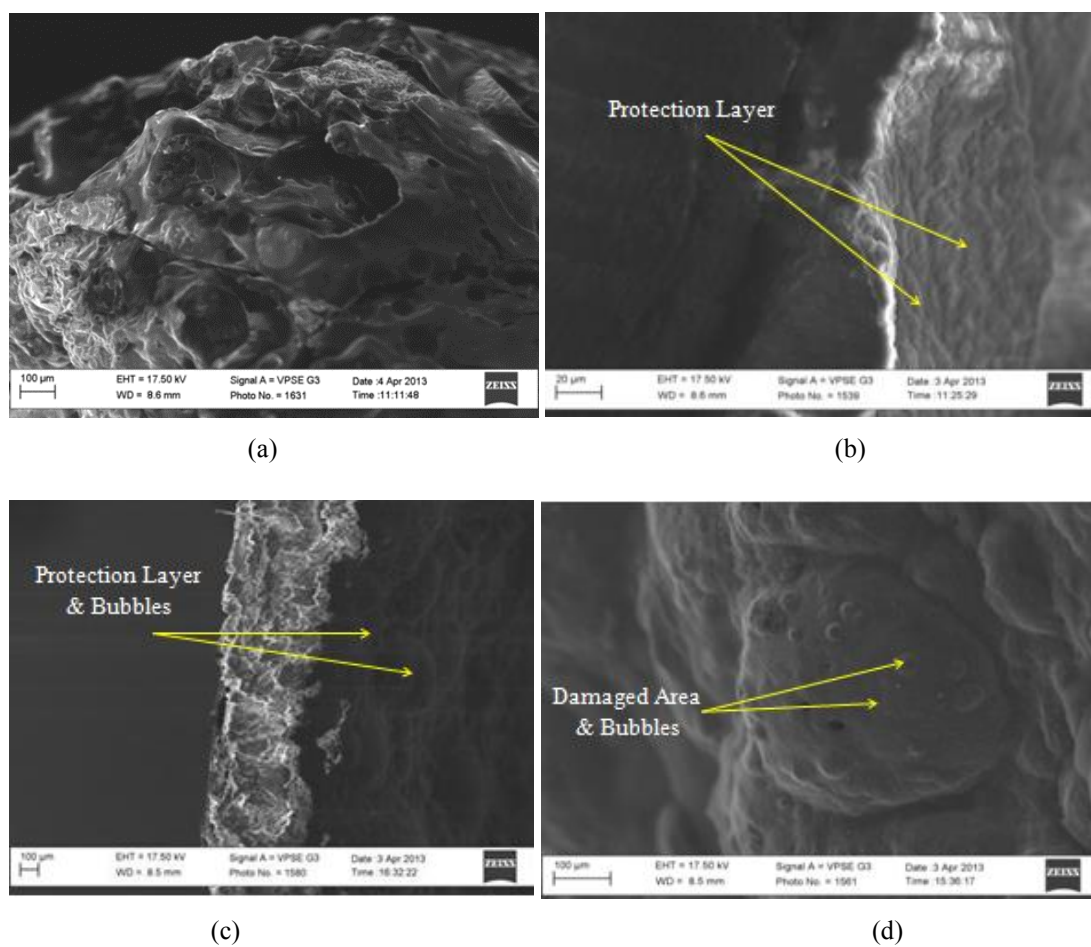| Specimen	V2	V1	V0
Pure PVC (Failed)			
5% Nanoclay		✓	
10% Nanoclay			✓
20% Nanoclay			✓
5% Nanotalc	✓		
10% Nanotalc	✓		
20% Nanotalc			✓
5% Graphene		✓	
10% Graphene			✓
20% Graphene			✓

(a)

(b)

(c)

(d)

Figure 2. SEM images of PVC samples: (a) pure PVC after burning, (b) burned PVC with 20 wt% nanoclay, (c) burned PVC with 20 wt% nanotalc, (d) burned PVC with 20 wt% graphene. Bar is 100 μm

Thermogravimetric curves exhibit the features of a polymer due to the unique sequence of the physiochemical reaction that occurs over specific temperature ranges and heating rates. The samples used for TGA contained a predetermined amount of the PVC nanocomposite samples.

Figures 3 to 5 show thermogravimetric curves of the PVC nanocomposites with different weight percentages of nanoclay, nanotalc, and graphene nanoflakes. A TGA study was employed to determine the weight-loss patterns of the prepared samples as a function of temperature. Measurements were started at room temperature (25°C) and ramped to 1,000°C at a heating rate of 10□C/min. A nitrogen purge of 50 mL/min and platinum HT sample pans were used.

The heat treatment of the PVC samples produced chemical reactions and process mechanisms, depending on the heating rate, medium, mass of polymer, and type and nature of filler materials. TGA curves of both pure PVC and PVC with different weight percentages of filler materials show a four-step weight loss. The thermogravimetric curve for pure PVC (Figure 3) appears to be quite dry since its weight is almost constant up to 100°C [15]. After 100°C, no significant weight loss was observed until irreversible decomposition began at 138°C. This indicates that pure PVC shows stability until 138°C. Weight loss in pure PVC occurs as a result of the decomposition process [16].

When the temperature reached 293°C, pure PVC displayed a drastic weight loss of around 60%, exhibiting the beginning of another irreversible decomposition where half of the PVC weight vanished [15]. At 311°C, separation of chlorine occurs, and finally cracking of the hydrogen back-bone takes place at 475°C. Pure PVC lost 92.25 wt.% of its initial weight at a temperature of 337.51°C, while PVC with 5 wt.% nanoclay lost 81.96 wt.% of its initial weight at 340.89°C. As can be seen in Figure 3, the PVC sample containing 10 wt.% of nanoclay lost 80.24% of its initial weight at a temperature of 364.87°C. The PVC containing 20 wt.% nanoclay provided better thermal stability compared to 5 and 10 wt.% clay and lost 74.38% of its initial weight at 338.38°C.
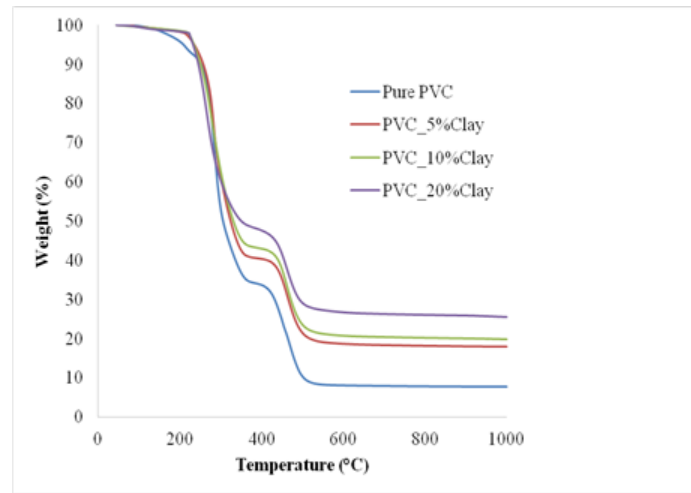
Figure 3. TGA analysis of pure PVC samples and PVC containing 5, 10, and 20 wt% nanoclay
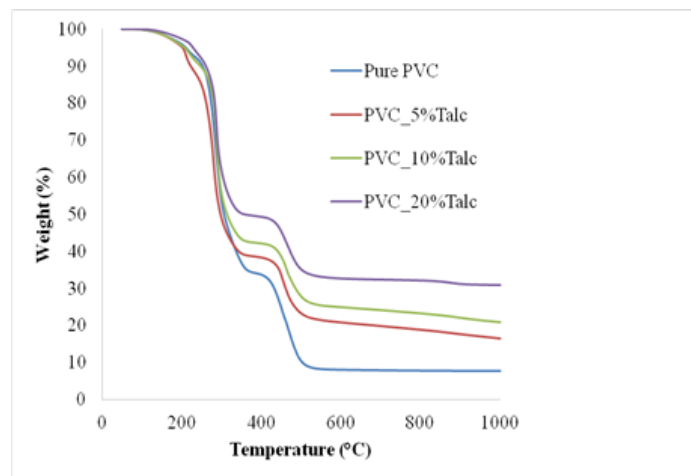

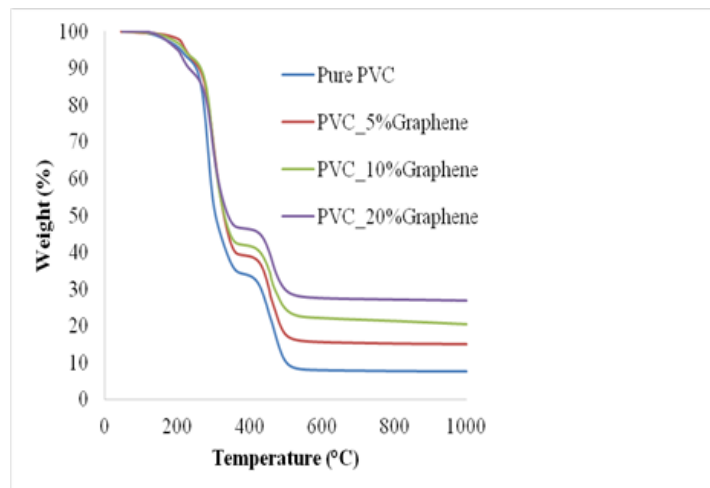**Figure 4**. TGA analysis of pure PVC samples and PVC containing 5, 10, and 20 wt% nanotalc

Figure 5: TGA analysis of pure PVC samples and PVC containing 5, 10, and 20 wt% graphene nanoflakes

These results show that as the weight percentage of nanoscale inclusions was increased, the thermal stability also increased.

Figure 4 shows the TGA analysis of the PVC samples with different weight percentages of nanotalc inclusions. As can be seen, the sample containing 5 wt.% nanotalc lost 83.54% of its initial weight at a temperature of 314.25°C, whereas the pure PVC sample lost 92.25% at a temperature of 337.51°C. The PVC containing 10 wt.% lost 79.11% of its initial weight at 310.40°C, while the PVC with 0% nanotalc lost 92.25 wt.% of its initial weight. The PVC containing 20 wt.% nanotalc exhibited greater thermal stability, with a weight loss of only 69.13% of its initial weight at 314.47°C.

As shown in Figure 5, pure PVC shows 92.25% weight lost at 337.51°C and the PVC sample with 5 wt.% graphene shows 84.79% weight loss wt.%at 366.75°C. The PVC with 10 wt.% graphene lost only 79.46% of its original weight at 348.98°C. However, the PVC containing 20 wt.% graphene shows higher thermal stability compared to the 5 and 10 wt.% of the same nanoscale inclusions. This sample lost 73.22% of its initial weight at a temperature of 352.30°C. The PVC with graphene inclusions exhibits significantly better thermal stability than polymer-only samples.

Differential Scanning Calorimetry Tests

Differential scanning calorimetry (DCS) is a technique to measure the thermal properties of polymers based on the rate at which they absorb heat energy compared to a reference material. In this test, DSC was used to determine the glass transition temperature (Tg) and degradation process of the PVC samples with different weight percentages of nanoscale filler materials. The DSC heat flow and temperature were calibrated with an indium standard prior to testing. The glass transition temperatures of the samples were determined in a nitrogen flow rate of 50 ml/min using DSC at a heating rate of 10°C/min. Table 2 provides Tg values of the PVC samples as a function of nanoscale filler material inclusions.

The Tg value of pure PVC is 55.98°C. The increase in Tg with the addition of nanoclay, nanotalc, and graphene nanoflakes may be attributed to the nanoscale filler materials and polar solvent (DMAC) entrapments in pores and voids, which could potentially cause dipole-dipole interactions with nanoscale filler materials and polar chlorine in the molecular structure. The Tg of the PVC nanocomposites tends to shift slightly towards the higher temperature compared to that of the pure PVC, indicating the change in polymer thermodynamics [23]. The Tg for the samples increases until there is 10 wt.% of nanofiller materials, and then it starts decreasing. These results indicate that a percolation threshold is around 10 wt.% of nanoscale fillers materials. The percolation threshold occurs at high nanofillers concentration, and in the absence of a conductive pathway, the polymeric materials are called insulators [10-12]. At a high wt.% of nanofiller materials, agglomeration occurs, owing to the lack of dispersion and interfacial interactions [11].

The Tg has been studied extensively for almost all polymers since it is one of their most fundamental

Table 2: Tg values of PVC samples as function of nanoscale filler materials.

Weight % of Nanofillers	Glass Transition Temperature (°C)
Pure PVC	55.98
5% Clays	80.92
10% Clays	81.14
20% Clays	61.94
5% Talc	74.45
10% Talc	87.36
20% Talc	52.86
5% Graphene	76.29
10% Graphene	76.20
20% Graphene	49.55

properties [13]. Generally, nanoscale filler materials directly affect the Tg of a polymer material, and the interaction of polymer chains with the surface of nanofiller material can alter the chain kinetics in the region surrounded by the nanofiller materials [13]. Strong interactions and possible covalent bonding between the polymer matrix and nanofiller materials most likely results in a higher Tg [13]. The reduction in Tg beyond 10 wt.% of the filler materials may be due to the presence of a higher concentration of filler materials, which may restrict the mobility of the polymer chain and aggregation/clustering of the filler materials [13,14]. The polymer degradation as measured by DSC appears as a distinct exotherm, as shown in Figures 6 to 9.
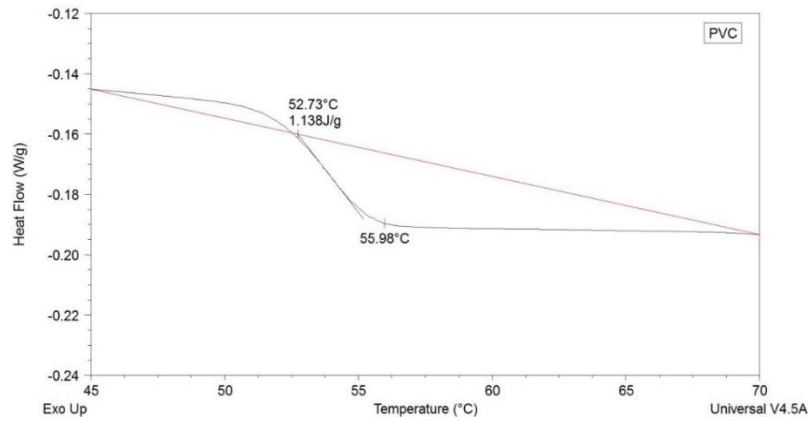

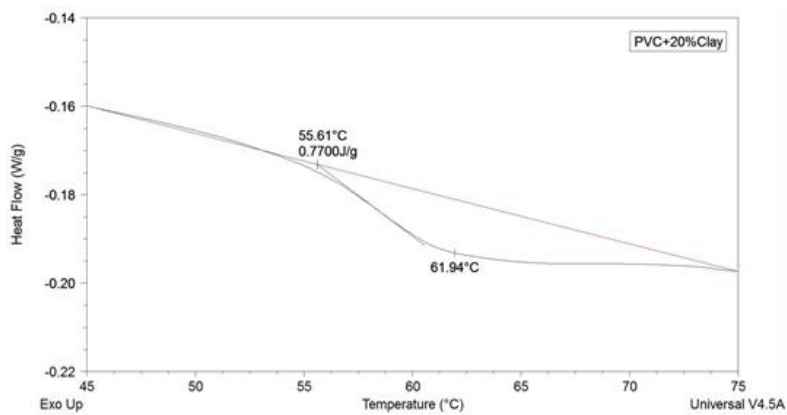**Figure 6.** DSC thermogram of pure PVC as function of time

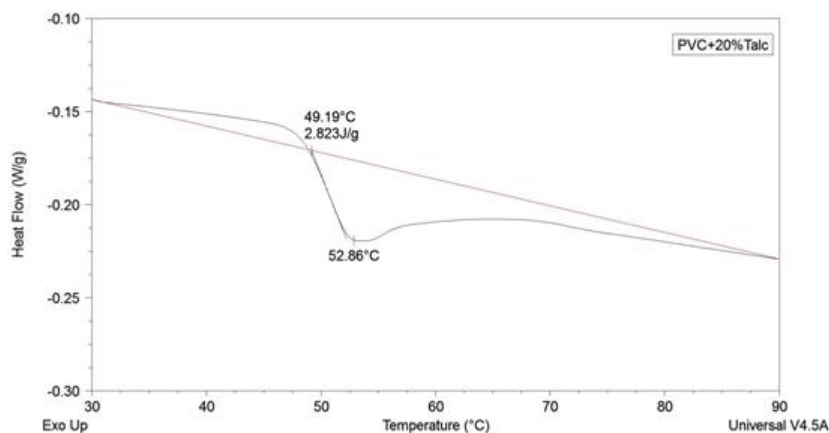**Figure 7.** DSC thermogram of PVC with 20 wt% nanoclay as function of time

Figure 8. DSC thermogram of PVC with 20 wt% nanotalc as function of time
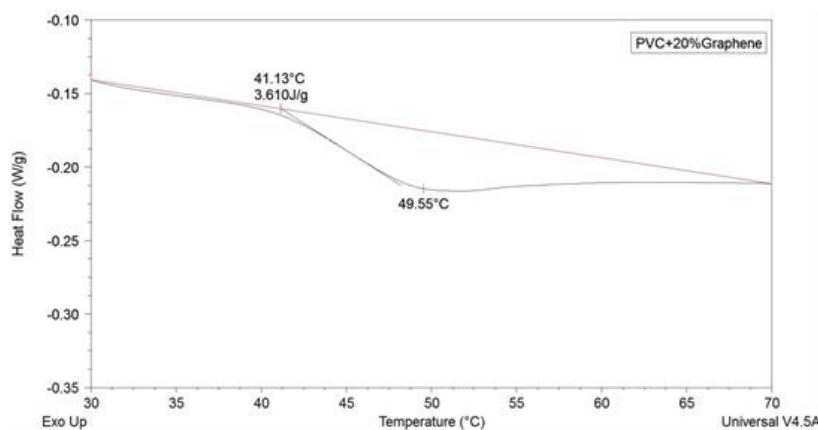
Figure 9. DSC thermogram of PVC with 20 wt% graphene nanoflakes as function of time

As can be seen in Figure 6, the transition observed between 52.73°C and 55.98°C reveals the Tg of pure PVC. The glass transition shifted to lower temperatures as the amount of nanofiller material in the sample was increased, except for the sample with 20 wt.% nanoclay, which exhibited a Tg between 55.61°C and 61.94°C. The reason for this unexpected shift in temperature was due to the processing conditions, orientation of nanoscale filler materials, and type and shape of the nanomaterials. DSC thermograms show that the range of glass transition temperatures (difference between the beginning Tg and the end Tg) widens as the concentration of nanofiller increases [17]. However, the sample with 20 wt.% nanotalc shows a reverse trend. Generally, the addition of nanofiller widens the relaxation temperature within the glass transition range. The specific heat accompanying the Tg decreases as the concentration of the filler material increases, which may be because of the increased molecular weight between entanglements, in addition to dilution of the PVC matrix that accompanies the increase in inclusion concentrations [17]. The influence of various nanoscale particles on the Tg of PVC samples has also been reported by other researchers [18-20].

PVC has been used in many industrial applications, but owing to its low thermal stability, its applications are limited for high temperature purposes. The reasons for adding nanoclay, nanotalc, and graphene nanoflakes are to improve its thermal stability and broaden its applications for various industries. Some studies show that good dispersion of nanoparticles in a polymer matrix could restrict the movement of polymer chain segments into higher glass transition temperatures [22-24]. Surface functionalization of nanoscale inclusions may drastically affect the Tg values of nanocomposite materials, which will be studied further.

Mechanical Properties of PVC Nanocomposites

Microtensile tests were conducted on samples having 20 wt.% nanoscale filler materials prepared for ASTM UL 94 studies. Table 3 provides the mechanical properties of the prepared samples. As shown, the Young's modulus, ultimate tensile strength, yield strength, and fracture stress of pure PVC samples were 603 MPa, 4 MPa, 3.9 MPa, and 2.5 MPa, respectively. In the presence of 20 wt.% graphene inclusions, these parameters of the PVC nanocomposite samples were increased to 775 MPa, 7.8 MPa, 9.3 MPa and 8.5 MPa, respectively. Similar results can be seen for other nanocomposites.

Mechanical properties of nanocomposite materials are mainly dependent on the homogeneous dispersion of nanoscale fillers in the matrix materials, surface functionalization, fabrication techniques and types, geometry, and orientation of the filler materials [18]. A lower concentration of filler materials acts as inactive materials distributed in the PVC matrix. As the concentration of filler materials increases, they can take part in load distribution and load sustaining load. These parameters also affect the fire-retardancy properties of PVC nanocomposite materials [25, 26].

As previously stated, most polymers can be decomposed or pyrolysed when exposed to heat, thus producing flammable volatiles that can eventually mix with air and ignite at high temperature [1]. When sufficient heat is generated, a self-sustaining combustion cycle can take place, resulting in further flame/fire. In order to increase fire-retardancy properties of polymeric materials, a number of different flame retardants, such as fluorine,

Table 3. Microtensile test results of prepared PVC samples with 20 wt.% nanoscale inclusions

Specimen	Young's Modulus (MPa)	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Fracture Stress (MPa)
Pure PVC	603	4	3.85	2.5
PVC + 20 wt.% Clay	538	4.7	4	4.2
PVC + 20 wt.% Talc	918	5.4	5.1	5.3
PVC +20 wt.% Graphene	775	7.8	9.3	8.5

chlorine, and boron, can be added. However, these materials can be costly, create environmental concerns, and increase the complexity of the process. The present study has indicated that by using nanoscale inclusions in these organic polymeric materials, their weakness could be significantly alleviated in different industrial applications, including those in transportation, packaging, electronics, energy, and defense. The nanomaterials here are considerably safe for producing different fire-retardant nanomaterials for those applications [27].

CONCLUSIONS

Improvement in the fire-retardant behavior of polymers is a major challenge for extending their life and use in several different industrial applications. In the present study, nanoscale inclusions, such as nanoclay, nanotalc, and graphene were incorporated with PVC, and the resultant nanocomposites were characterized using the ASTM UL 94 standard test, TGA, DSC, SEM, and microtensile test units. Test results showed that the fire retardancy, thermal stability, and mechanical properties of PVC nanocomposites were significantly increased with the nanoscale inclusion contents. Especially, in the presence of graphene, physical properties of the PVC nanocomposites were much higher when compared to other inclusions, which may be because of the extraordinary physical properties of graphene nanoflakes. This study may open up new possibilities of using many nanoscale inclusions in various polymers as flame-retardant materials for different industrial applications in the near future.

ACKNOWLEDGMENT

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