Chapter 4:

Poly(VAc-*t*-BA-t-AA) terpolymer/kaolin clay composites and their characterizations

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ABSTRACT

In this study, the preparation and characterization of poly(vinyl acetate) (PVAc) based terpolymer/kaolin clay composites are described. Conventional free-radical emulsion polymerization was used to prepare a terpolymer of vinyl acetate (VAc), butyl acrylate (BA), and acrylic acid (AA). ^{1}H NMR analysis confirmed the successful preparation of the terpolymer. The conversion of the monomers was calculated gravimetrically and found to be 90 % after one hour of polymerization time. Different amounts of kaolin clay were added to the terpolymer latex and were mixed to prepare the terpolymer/kaolin clay composites. NMR, FT-IR, TGA, DSC and FESEM analyses were used to characterize the terpolymer and terpolymer/clay composites. Mechanical properties such as tensile strength, modulus, and elongation at break of the prepared terpolymer/clay composites were also evaluated. A rubber process analyzer (RPA) was used to study rheological properties of the polymer composites. All the analyses establish the presence of strong clay-polymer interaction in the composite for which the prepared composites exhibit improved thermal properties.

KEYWORDS: Vinyl acetate, Emulsion polymerization, Clay composite, FTIR, RPA.

4.1. INTRODUCTION

In recent years, the research on the synthesis of inorganic-organic polymer hybrid materials such as layer silicate/polymer nanocomposites have attracted great interest due to their wide superior physical, mechanical and thermal properties compared to the other composites.[127,128,14] A composite is a microscopic blend of two or more polymeric, metallic, or ceramic materials that are linked together in specific ways.[26] Clay composites (organic/inorganic hybrid materials) are made up of clay intercalated with a liquid polymer, with the intercalation group consisting of aluminosilicates of the smectite group.[101] Clays are naturally occurring silicate minerals that are readily available. This has led to their widespread use in novel chemical processes, owing to the low cost and environmentally friendly nature of the material. Few studies have described how different clays, such as montmorillonite, mica, hectorite, bentonite, or laponite, can be used to stabilize emulsion colloidal latex using the emulsion polymerization technique and clay minerals colloidal latex.[61,62] Sheeted of stable aqua silicates of aluminum, iron, and magnesium, sheeted tape, and mixed-sheeted structures are found in the structure of the clays. Clay has a high dispersity, hydrophilicity, sorption ability, ion exchangeability, and the highest tendency to adsorb various organic compounds due to this structural feature.[129] Kaolin is a clay mineral commonly used for filling and paper coating.[57] With the chemical formula $Al_2Si_2O_5(OH)_4$, it is a one-of-a-kind phyllosilicate mineral. One tetrahedral SiO_4 silica is linked through an oxygen atom to one octahedral $Al_2(OH)_4$ alumina plate in the 1:1 di-octahedral structure of kaolin clay.[58] Kaolin contributes to improved paper appearance and printability by improving properties such as gloss, smoothness, brightness, and opacity.[130] The use of a suitable coupling agent to modify kaolinite can result in good filler particle compatibility and dispersibility in a specific polymer matrix.[77] The research field of polymer/clay nanocomposites begin from after the report on the nylon-6/clay nanocomposites by the Toyota research group.[94,95] Polymer/clay nanocomposites have good properties like flexibility and moldability, and also with the strength and heat stability.[28] Clay improve reinforcing effect the polymer matrix.[131,106]

The intercalation of polyvinyl acetate (PVAc) with clay improved the composite properties, and it has some interesting applications. Compared to pure polyvinyl acetate, Mohsen-Nia et al. found that PVAc/montmorillonite nanocomposites have a higher glass transition temperature and thermal stability.[106] Mrah et al. investigated polymer/Maghnite nanocomposite have good improvement of thermal stability, mechanical and electrical (electrical conductivity increases) properties which are useful in materials in flexible electronics and in conductive coatings.[132] Rubber/poly (ethylene-*co*-vinyl acetate) nanocomposite materials maximum mechanical properties and the lowermost loss factor, which are applicale as reuse of tire in the rubber industry.[133] Recently quantum dots/ethylene VAc copolymer composite films for lighting applications and flexible displays were reported by Li et al.[109] Poly(ethylene-*co*-VAc) composites with silver-calcined scallop shell powder have good antibacterial properties and could be used in food packaging and biomedical sample storage.[111] The vinyl acetate is a superiorly green monomer, and their copolymers prepared using various methods have a variety of applications,[59,134-136] indicating the potential eco-friendliness of this study of a novel type of terpolymer composites.[137] The kaolin clay was chosen because of its non-toxicity, easily available, and also affordable. It may be used for the enhancement of filler particle compatibility and dispersibility in a particular polymer, better biodegradation nature and enhance the barrier properties. Kaolin enhances the barrier properties for water vapor. Kaolin increase the biodegradation nature of polymer/clay composites. That makes polymer/clay composites more suitable as film.[138] In this study, emulsion polymerization is used for the synthesis of terpolymer which is a simple and environmentally friendly experiment.[137]

In recent times, the effort has been made to progress on the preparation of tailor-made polymer-clay composites that illustrate the random distribution of the single-layered silicate and offer additional control over the polymer design.[139] If the monomers are chosen carefully then terpolymers of vinyl acetate overcome some disadvantages of homopolymer which make it unique. As a result of the prospectus of various applications and the green aspect of using vinyl acetate monomer, future research in this field is of great interest.

In this study, we used emulsion polymerization to make poly(VAc-*t*-BA-*t*-AA) terpolymers and five batches of polymer/clay composites were made using different clay loading. To the best of our knowledge, this is the first account in the study of the effect of kaolin clay on poly(VAc-*t*-BA-*t*-AA) terpolymers. The composites were characterized using various analytical techniques such as ^{1}H NMR, FT-IR, DSC, TGA and SEM. Tensile testing was used to evaluate the mechanical properties of the terpolymer composites. RPA was employed to investigate the rheological behavior of the polymer/clay composites. The synthesis terpolymer/clay composites show better modulus or reinforcement properties than other copolymer/clay composites.

4.2. PREPARATION

4.2.1. Synthesis of terpolymer

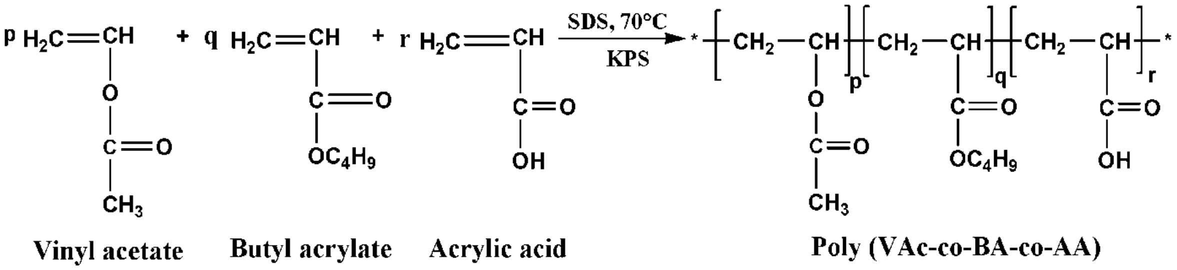
In a typical terpolymerization reaction, distilled water (164g, 9.11mol) and the surfactant SDS (3.28g, 11.37mol) were combined in a dry round bottom flask (RB) with a stirrer, magnetic bar, and rubber septum. After 15 minutes of stirring, different compositions of VAc/BA/AA monomers (total 82 g) were injected into the RB. After another 30 minutes of stirring, initiator KPS (0.656 g, 2.42 mmol) was added to the reaction mixture and stirred for an hour. After that, the RB was sealed, and nitrogen gas was passed through the mixture for 60 minutes to remove any dissolved oxygen. Then the reaction RB was placed in a preheated oil bath with a reflux condenser and the polymerization reaction was allowed to run for 60 minutes at 470 rpm at 70°C. The conversion was determined gravimetrically after the aliquot was removed at the end of the polymerization.

4.2.2. Synthesis of terpolymer/kaolin clay composites

The prepared terpolymer latex was stirred at 540 rpm for 24 hours at room temperature to obtain a uniform latex. Filtering the solution separates the coagulum from the solution, and the coagulum percentage was calculated to be 3.2 percent. The stable latex was then mixed with kaolin clay in various ratios of 5%, 10%, 15%, and 20% respectively with respect to latex. After 36 hours of stirring with 540 rpm, the latex mixtures were transferred to a glass petri dish. To make the uniform film, the latex mixtures were dried for two days at 60°C in an oven. NMR, FTIR, DSC, and TGA analyses were performed on the obtained films (terpolymer/clay composites). The prepared terpolymer/clay composites were also evaluated for mechanical properties such as tensile strength, modulus, elongation at break, and hysteresis. The RPA was used to assess the rheological properties of the polymer composites.

4.3. RESULTS AND DISCUSSION

The poly(VAc-*t*-BA-*t*-AA) terpolymer (Scheme 4.1.) of VAc, BA, and AA monomers was prepared by FRP using an emulsion polymerization technique. The reaction was carried out at 70 °C for 60 minutes at 470 rpm. For the synthesis of terpolymer latex, the KPS initiator was combined with the SDS surfactant in an emulsion polymerization process. The monomer conversion for the polymerization was determined by gravimetric analysis and was found to be 90% after 1 hr. The latex was found to be stable even after few weeks of storage as evident by UV-Visible analysis. The molecular weight of the terpolymer was determined by GPC analysis and was already reported in our earlier research publication.[137] The number average molecular weight for this terpolymer was found to be 100,217 g/mol. Various amounts of kaolin clay were added to this terpolymer latex to prepare the polymer/kaolin clay composites. Table 4.1. summaries the compositional breakup of the ingredients involved in making the polymer/kaolin clay composites.



Scheme 4.1. Terpolymerization of vinyl acetate, butyl acrylate and acrylic acid.

Table 4.1. The details of the preparation of the polymer/kaolin clay composites.

Sample name	Polymerization system	Mole ratio	Conversion %	Polymer latex(g)/ kaolin clay (g)	% of Kaolin clay
*V-1	VAc/BA/AA	0.15/ 0.51/0.045	90	55/0	0
V-2				45/2.25	5
V-3				45/4.5	10
V-4				45/6.75	15
V-5				33/6.6	20

* V-1 indicate for poly(VAc-*t*-BA-*t*-AA) and V-2, V-3, V-4, V-5 for poly(VAc-*t*-BA-*t*-AA)/clay composites with 5%, 10%, 15%, 20% respectively.

4.3.1. Nuclear magnetic resonance spectrometry (^{1}H NMR) analysis

The incorporation of the monomers was confirmed by the ^{1}H NMR spectrum. The ^{1}H NMR spectra of poly(VAc-*t*-BA-*t*-AA) and poly(VAc-*t*-BA-*t*-AA)/clay composites are shown in Figure 4.1. and 4.2 respectively. In Figure 4.1., the signal at δ =0.96 (a) and 1.99 (f) ppm are attributed to the methyl protons of BA and VAc respectively. Signals at δ =1.39, 1.62 and 4.06 ppm are assigned to the protons of —CH_2— (b), —CH_2— (c) and —O—CH_2— (d) of BA respectively. Signals at δ =1.92 and 2.36 ppm correspond to the backbone protons of —CH— (e) and —CH— (g) of BA and AA respectively. The ^{1}H NMR spectra of poly(VAc-*t*-BA-*t*-AA)/clay composites with different kaolin clay ratios was shown in Figure 4.2.

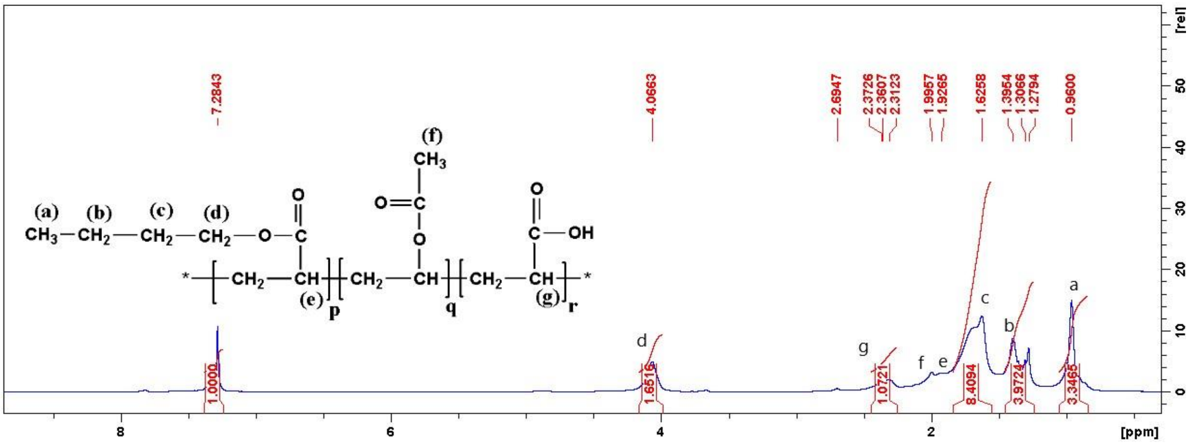


Figure 4.1. ^{1}H NMR spectrum of poly(VAc-*t*-BA-*t*-AA) terpolymer.

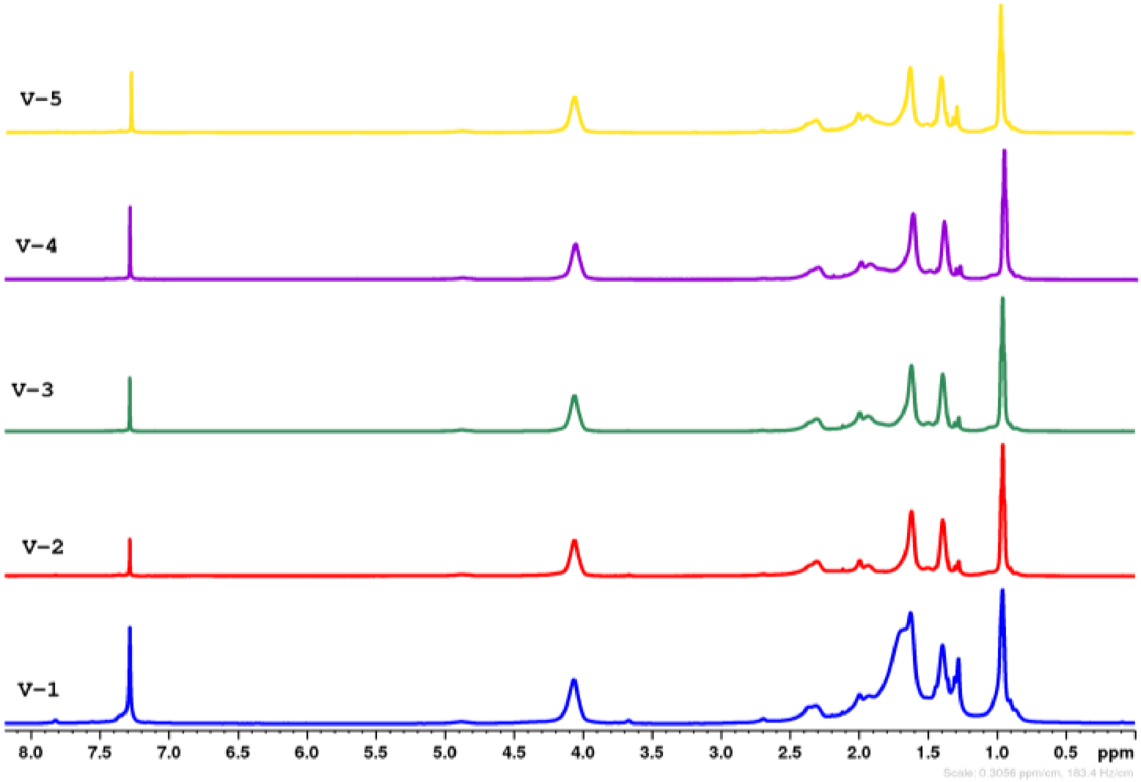


Figure 4.2. ^{1}H NMR spectra of poly(VAc-*t*-BA-*t*-AA)/clay composites.

4.3.2. Fourier transform infrared spectroscopy (FTIR) analysis

The FT-IR spectra for the carbonyl group absorption of terpolymer in the presence of different clay concentrations, t_c, was shown in Figure 4.3.(a). In this spectrum, it was seen that as t_c increases, the absorption band position of >C=O stretching shifts to a lower frequency range (by 1 cm^{-1} in case of 5%, 2 cm^{-1} in case of 10%, 3 cm^{-1} in 15%, and 4 cm^{-1} in 20%). Figure 4.3.(b) shows that with increasing clay amount, Al—O stretching frequencies of kaolin clay shifted to a higher frequency (473.83 cm^{-1} in case of 5% clay, 476.34 cm^{-1} in case of 10% clay, 477.06 cm^{-1} in 15% clay, 478.85 cm^{-1} in 20% clay). It means that when the >Al=O group of the clay layers interacts with the >C=O group of the acrylate monomer ($>C^{\delta-}=O^{\delta+}\cdots\cdots{}^{\delta-}Al=O$) (Scheme 4.2), the double bond nature of the carbonyl group is reduced, and the absorption band frequency decreases.[138] Figure 4.3.(a) shows C—H stretching of polymer composites at 2961, 2960, 2959, 2958, and 2957 cm^{-1}.[101] In comparison to pure polymer, the C—H stretching band of polymer composites shifted towards lower a frequency (by 1 cm^{-1} in case of 5%, 2 cm^{-1} in case of 10%, 3 cm^{-1} in 15%, and 4 cm^{-1} in 20%). This refers to the adsorption of polymer and kaolin clay particles via hydrogen bonding between the Si—O—H (silanol) of clay and the polymer molecules.[139] Figure 4.3.(b) shows the bands at 1061 and 1070 cm^{-1}, which correspond to the Si—O stretching frequencies of kaolin clay. Similarly, the shift in Al—O—Si stretching frequencies of kaolin was observed at 536, 533 cm^{-1}.[140] The frequency of Si—O groups in kaolin is slightly reduced as a result of polymer molecules interacting with clay particles.[139]

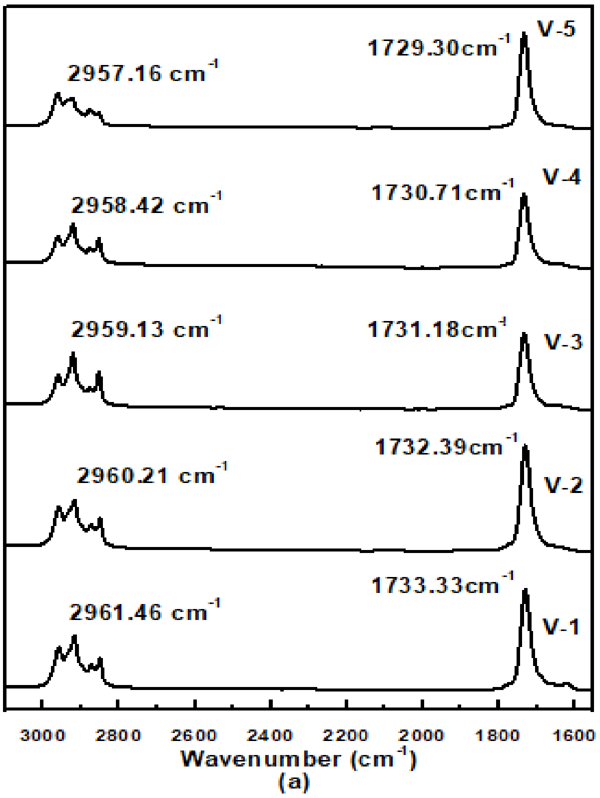


Figure 4.3. (a) FTIR spectra of terpolymer and terpolymer/clay composites in absorption.

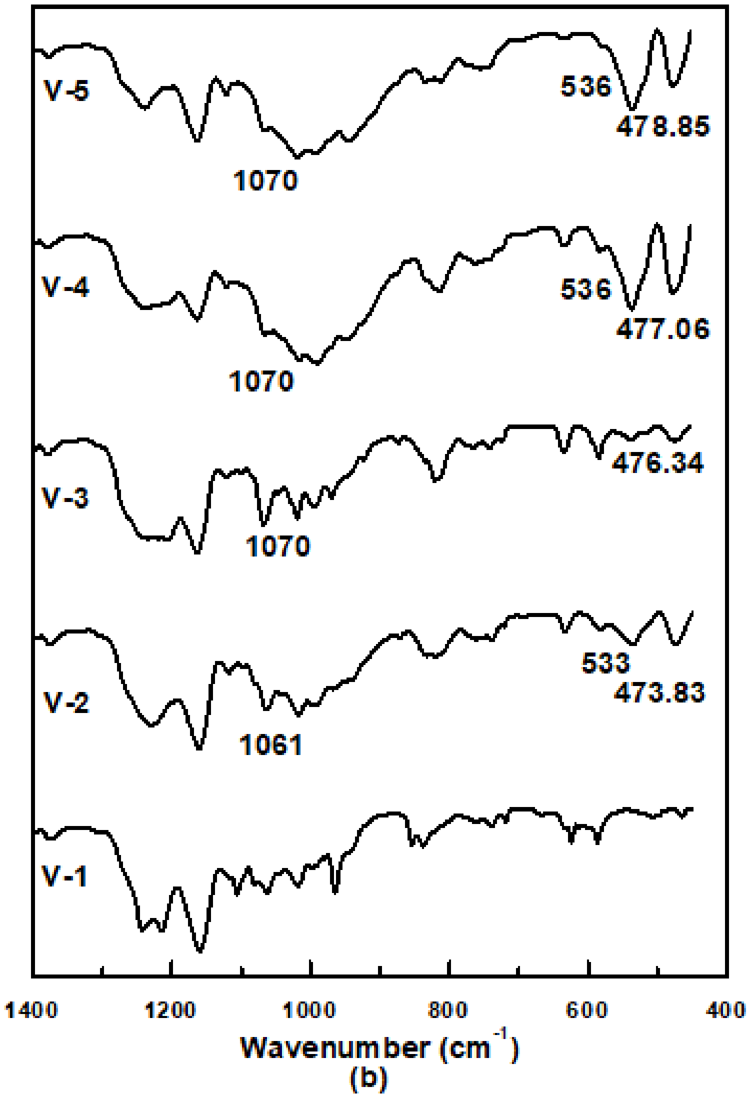
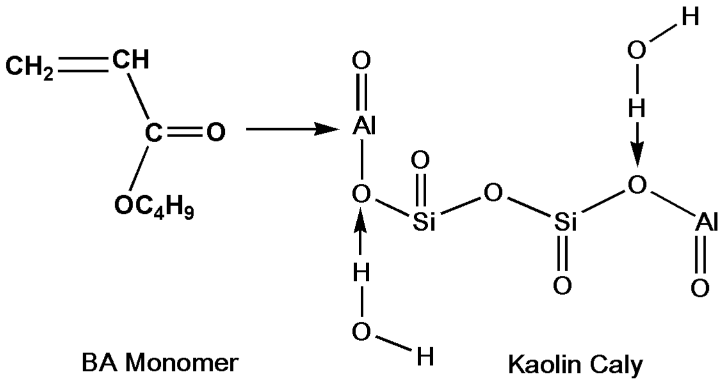


Figure 4.3.(b) FTIR spectra of terpolymer and terpolymer/clay composites in absorption.



Scheme 4.2. Schematic presentation of chemical interaction of >C=O groups and –Al=O groups of BA and clay respectively.

4.3.3. Differential scanning calorimetry (DSC) analysis

The T_g of the terpolymer/clay composites was determined using DSC measurements. The T_g values of V-1, V-2, V-3, V-4 and V-5 terpolymer/clay composites were found to be −42, −41, −40, −39, and −38°C respectively, as shown in Figure 4.4. and the same also have been summarized in Table 4.2. With an increase in the amount of clay, the T_g values shifted toward higher temperatures. The presence of clay in the polymer matrix decreases the free segmental motion of the polymer chains and hence increases the glass transition temperature.[128] The reported T_g of polyvinyl acetate/ montmorillonite nanocomposite is 32.2°C to 36.2°C,[110,106] which is much higher than the T_g of terpolymer/clay composites prepared in this work. Low T_g indicates the amorphous nature of the composites.

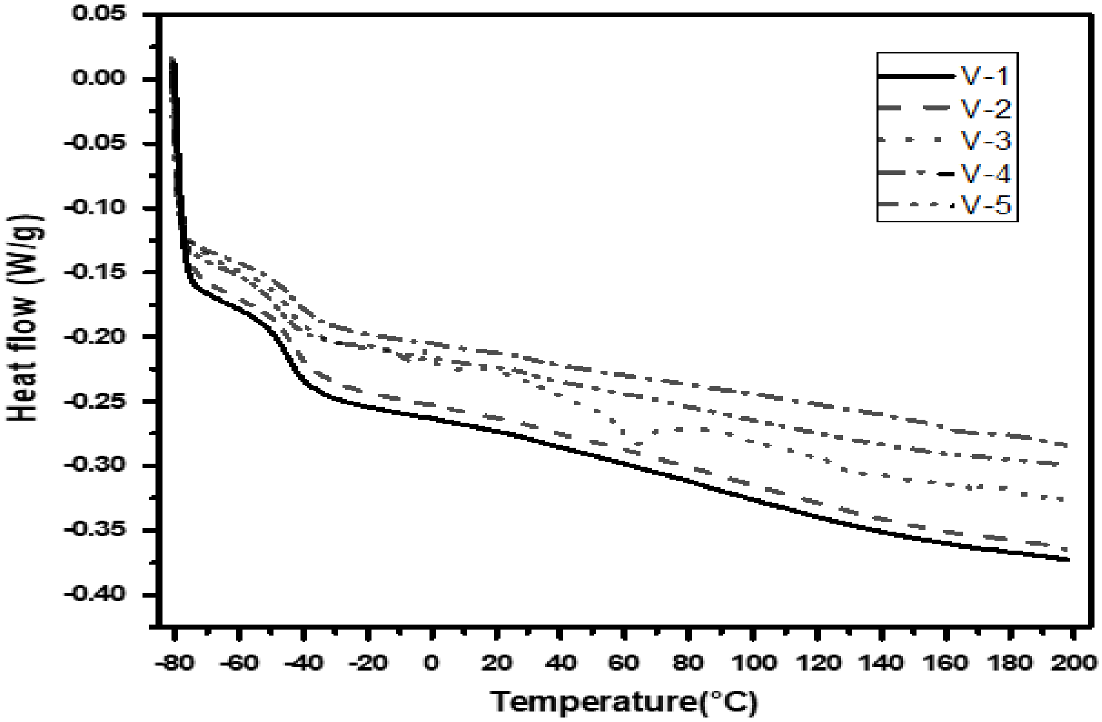


Figure 4.4. DSC plots of terpolymer and terpolymer/clay composites.

4.3.4. Thermogravimetric analysis (TGA) analysis

The thermal stability of the terpolymer/clay composites was investigated using TGA. The different TGA and Derivative thermogravimetry (DTG) curves of terpolymer and terpolymer/clay composites are shown in Figures 4.5.(a) and 4.5.(b). The degradation temperature of terpolymer and terpolymer/clay composites is summarized in Table 4.2. It shows the terpolymer/clay composites have higher thermal stability than pure terpolymer. The stability of polymer clay composites improves as the amount of clay in the composites increases. This is due to the chemical attachment of the kaolin clay particles to the polymer chains.[128] Batistella et. al studied ethylene-VAc/kaolinite composites and observed a two-stage degradation in TGA curves,[141] whereas, in this work, a single-stage degradation pattern is observed. The T_{max} values (highest is 540°C) reported in this work are also comparatively higher than the values reported by Batistella et. al.

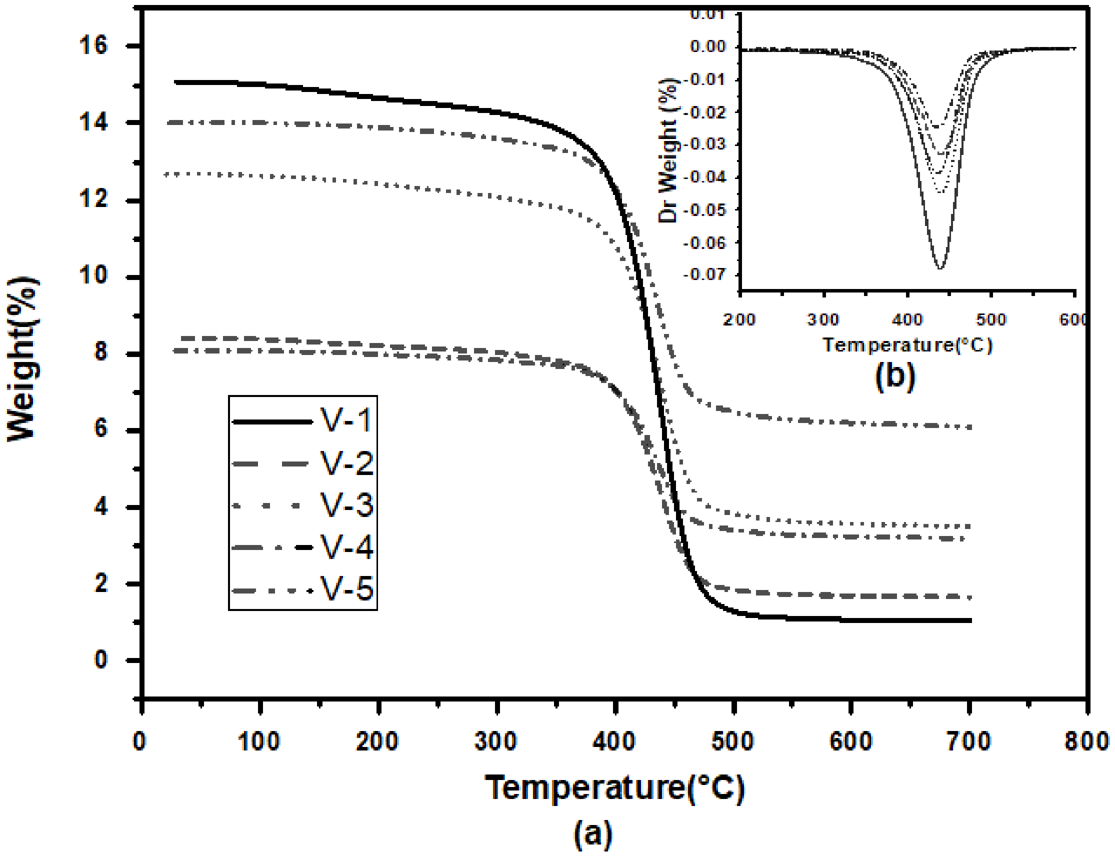


Figure 4.5. (**a**) TGA and (**b**) DTG plot of terpolymer and terpolymer/clay composites.

Table 4.2. Thermal properties of terpolymer and terpolymer/clay composites.

Sample	Kaolin loading (%wt w.r.t. polymer wt)	T_{onset} (°C)	T_{max} (°C)	T_g (°C)
V-1	Nil	293	508	-42
V-2	5	314	518	-41
V-3	10	329	523	-40
V-4	15	331	527	-39
V-5	20	338	540	-38

4.3.5. Physico-mechanical properties of terpolymer/clay composites

Mechanical properties such as tensile strength, modulus and elongation at break of the terpolymer have been listed in Table 4.3. Table 4.3. shows that in case of composites, the reinforcement of clay in terms of tensile modulus at 50%, 100%, 200%, and 300% elongation increased with increasing the surface area of the kaolin clay (or increase of the percentage of kaolin clay) in the compositions up to V-4, after which it decreased with further increase of clay loading. The tensile strength of the composites decreased as the amount of kaolin clay in the mixture increased at very high extend. The strength of the composites was reduced as a result of clay aggregation at very high clay loading.[142] Elongation at break decreased as the percent of kaolin clay (or the surface area of composites) increased up to V-3, after which it decreased (in the composite V-5). This is due to the improved polymer reinforcement and internal plasticizing effect imparted by the clay present in the polymer whereas elongation was reduced due to the aggregation tendency of clay at higher clay loading.[143] The reinforcement parameter refers to the physico-mechanical properties of the materials. As a result, as the amount of clay in the composites increases, the physico-mechanical properties of the polymer increases.

Table 4.3. Physico-mechanical properties of terpolymer and terpolymer/clay compositions

Sample name	a_0 (mm)	b_0 (mm)	Modulus at 50% (MPa)	Modulus at 100% (MPa)	Modulus at 200% (MPa)	Modulus at 300% (MPa)	Tensile strength (MPa)	Elongation at break (%)	Wa (Nm)	F-max (MPa)
V-1	2.52	3.15	0.065	0.098	0.175	0.065	1.290	962.6	1.43	1.290
V-2	2.14	3.00	0.122	0.187	0.258	0.122	0.830	821.8	0.81	0.832
V-3	1.20	3.00	0.139	0.187	0.295	0.139	0.211	508.8	0.19	0.440
V-4	0.85	3.00	0.175	0.187	0.189	0.175	-	-	-	0.193
V-5	1.30	3.00	0.170	0.171	0.166	0.170	0.050	995.1	0.17	0.175

4.3.6. Hysteresis test

The hysteresis loss of polymer compounds is compared in Figure 4.6 with the thickness (1.3mm- 2.30mm) and width (3mm) of composite films. The hysteresis loss, also known as the Mullins effect is a stress softening technique. The Mullins effect describes the amount of energy generated when a cyclic deformation occurs. In terms of viscoelastically and hydrodynamically, the hysteresis of polymer composites increases as the amount of clay or the specific surface area of clay increases. The different specific surface area of polymer/clay blends has a clear impact on clay minerals.[143] The amount of hysteresis loss-area under the curve was calculated using Origin 8 computer software. Meneghetti et al. reported that the hysteresis loop created by short glass fiber and polypropylene increased with the number of cycles.[144] Figure 4.6. shows that cyclic energy loss is lower in the first cycle than the second cycle and that the fourth cycle has the highest hysteresis energy loss. The hysteresis loss increased with the number of cycles in the loop, indicating that energy losses increased with the number of cycles in the loop. This is clearly due to the low filler–filler or terpolymer-clay interaction that takes place at a higher specific surface area filled polymer compound with the increase in the number of cycles. This indicates that the energy dissipation of the polymer composite with clay was more effective through the loading and unloading cycles. This could be due to the reason of destruction and delayed restoration of the interactions among the clay particles and the polymer at different strain amounts during the cycles.[145]

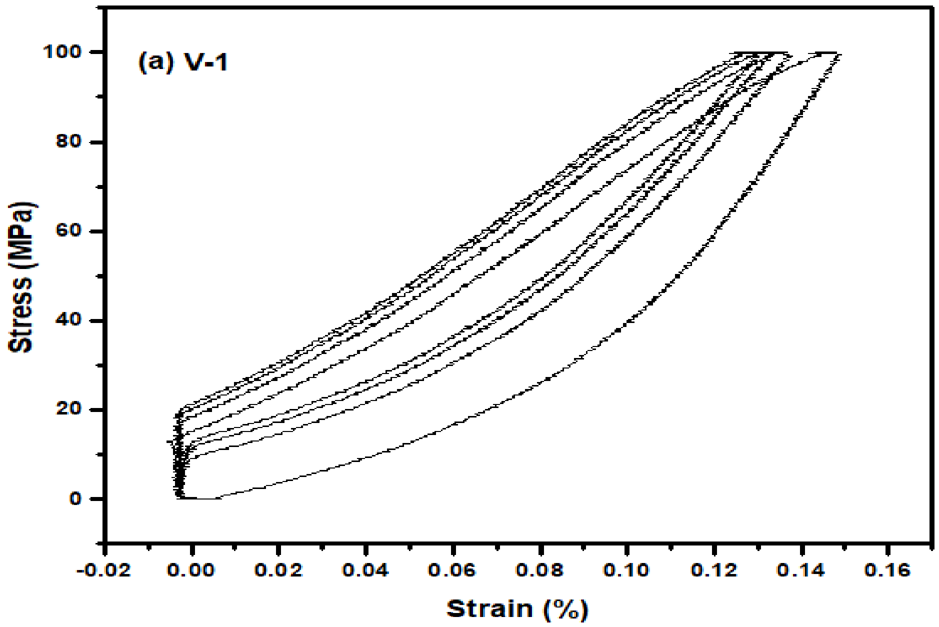


Figure 4.6.(a) Hysteresis loops for terpolymer composites V-1.

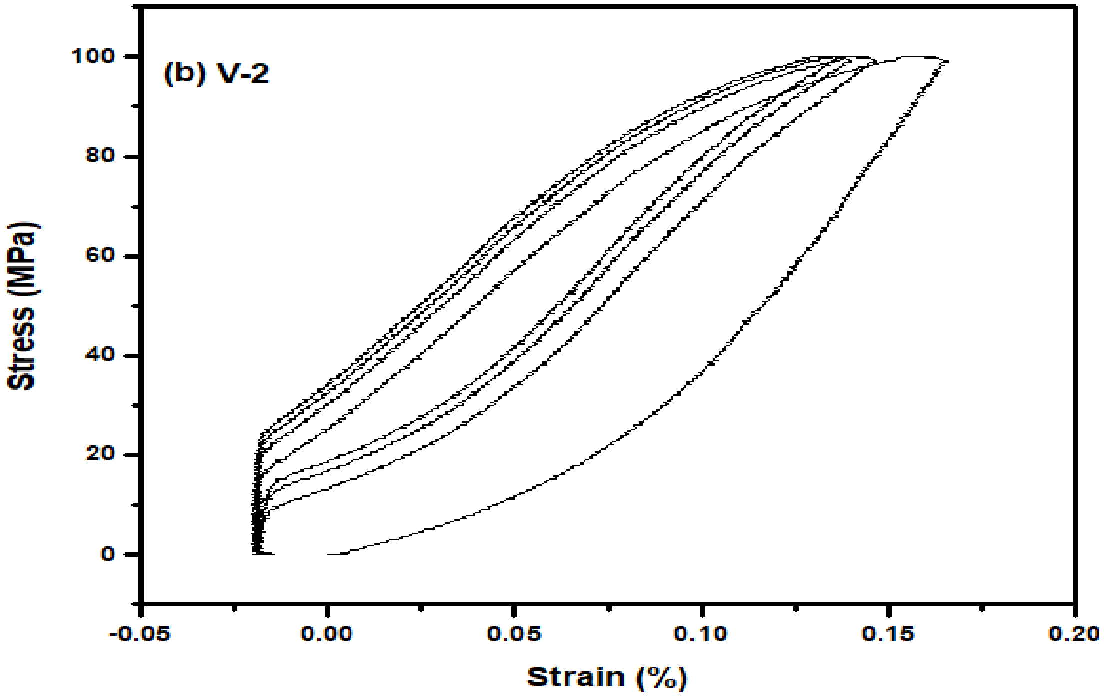


Figure 4.6.(b) Hysteresis loops for terpolymer/clay composites V-2.

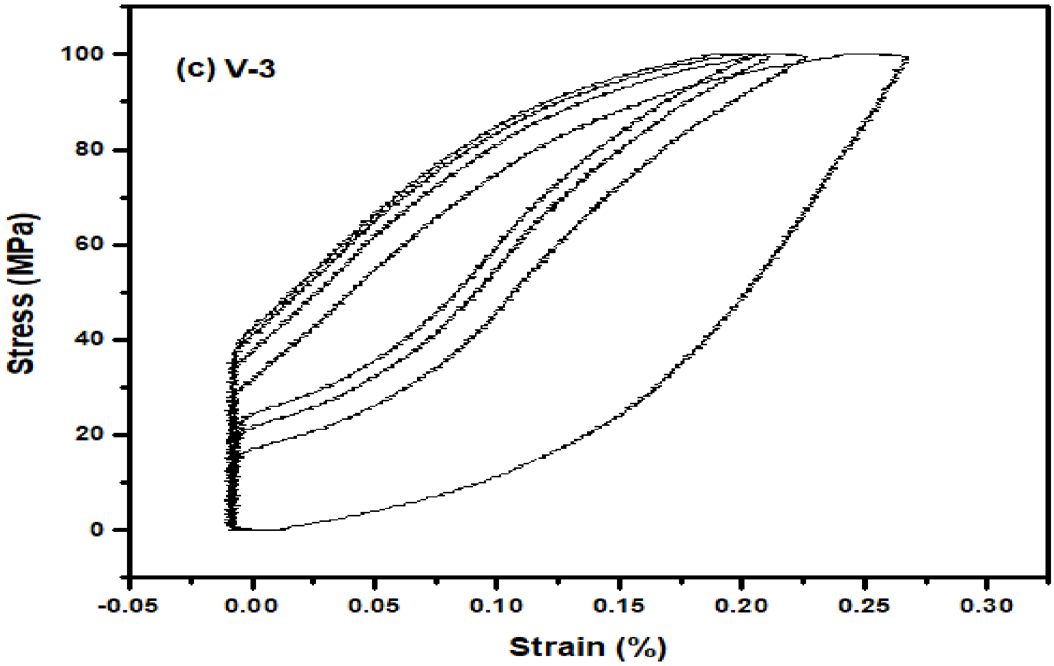


Figure 4.6.(c) Hysteresis loops for terpolymer and terpolymer/clay composites V-3.

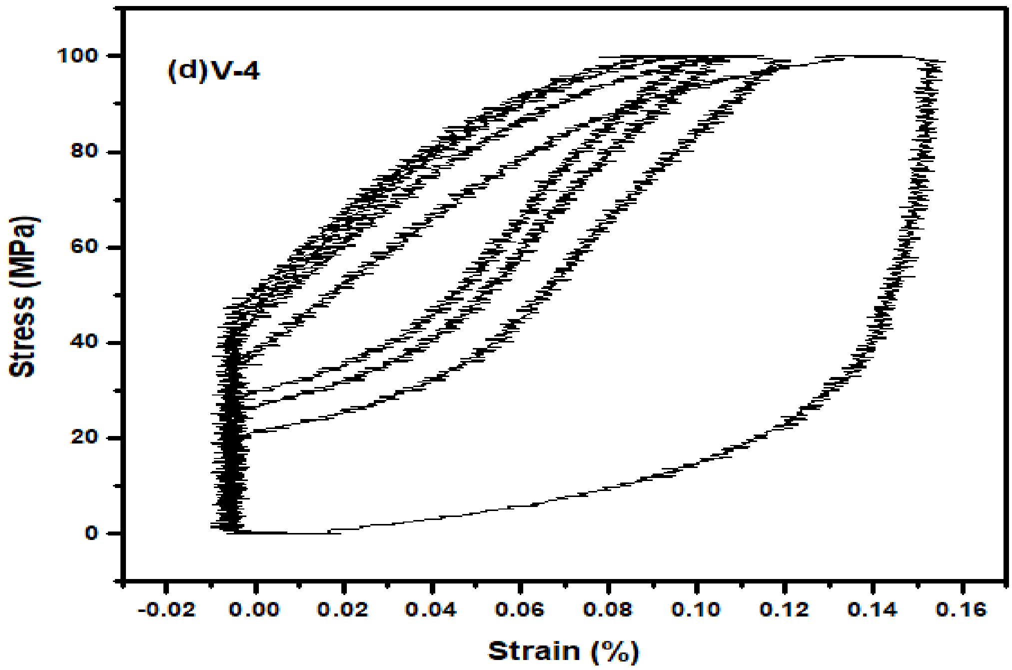


Figure 4.6.(d) Hysteresis loops for terpolymer and terpolymer/clay composites V-4.

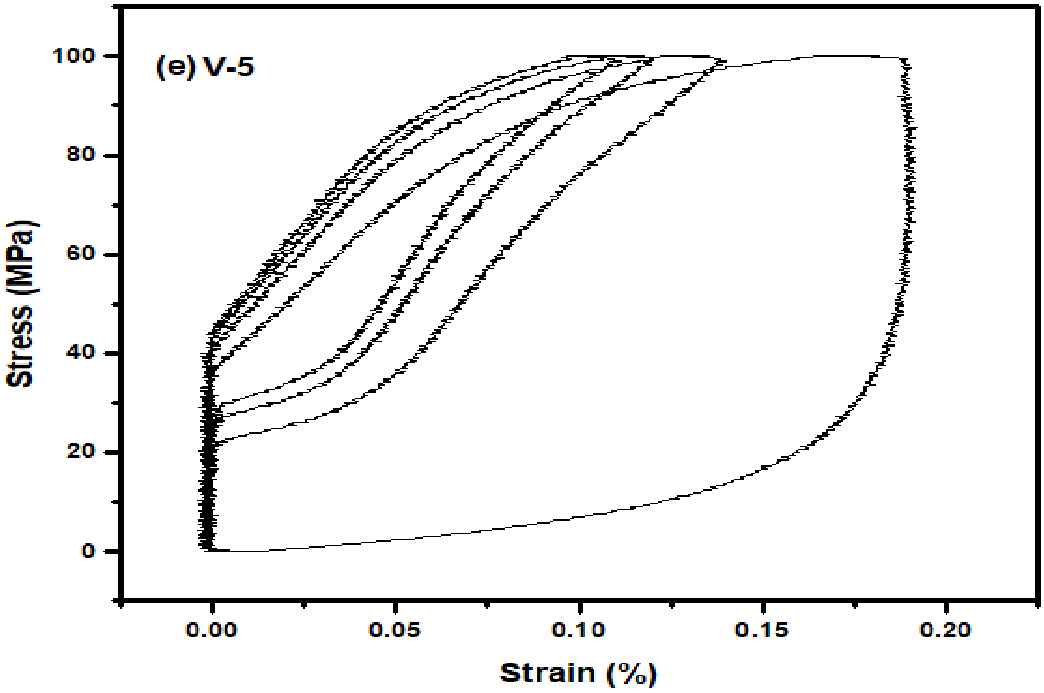


Figure 4.6.(e) Hysteresis loops for terpolymer and terpolymer/clay composites V-5.

4.3.7. Rheological properties of terpolymer/clay composites analysis

Using an RPA, the melt rheological properties of the polymer composites were assessed. Dynamic viscoelastic properties of the polymer composites were characterized by storage modulus (G′), loss modulus (G″) and loss factor (tan δ = G″/G′) of the terpolymer and its clay composites under strain sweep and are shown in Figures 4.7.(a), 4.7.(b) and 4.7.(c). Figure 4.7.(a) depicts that terpolymer/clay composites storage modulus (G′) curve which shows a non-linear viscoelastic behavior, and the modulus decrease with enhancing strain. This demonstrates that there was a structural breakdown. According to Datta et al. in the studied of (S–B–S) copolymer of vinyl-styrene, butadiene and styrene, the modulus decreases with enhancing strain. This is mainly at temperature rises to 120°C, when subjected to increase strain then causes the uncoiling of polybutadiene units quickly and the fragmentation of polystyrene units into smaller pieces.[146] This is similar to what we found in our research. With increasing strain percent, there is less confrontation in both elastic deformation and viscous flow due to these changes. As a result, the curves showed a sigmoidal decrease in the character.[146] The storage modulus reduction was caused by increasing clay amounts in the composite samples. This may be due to the filler influence of clays and successive interactions with polymer chains in these composites. In fact, low strain percent the storage modulus was high for high clay amount which features to well dispersion of clay in the matrix of the polymer. The outcome is useful to expect the capacity of dispersion of clay in the matrix of polymer composites. The curve of loss modulus (G″) versus strain percent is shown in Figure 4.7.(b). The loss modulus decreases as the strain percent increases because the materials crack into smaller units. Datta et al. also reported that the loss modulus of (S–B–S) copolymer of vinyl styrene, butadiene and styrene decreased at a certain point with increased strain, due to the materials cracking into slighter units. The approximately same type of findings can be found in our polymer composites.[146] As a result, composites exhibit a flow nature. However, the enhancement of loss modulus in the low-frequency region with an increase in the concentration of clay. This performance and magnitude of modulus development may be due to more interaction of clay and polymer chains because of additional effective dispersion given by clay. Figure 4.7.(c) shows the plot of loss factor (tan δ) versus strain % for terpolymer/clay composites which shows that as the clay content increased, the strain% and the tan δ also increased. The improved flow behavior (or enhancement of viscous property) in the direction of the shear strain helped by the polymer composites in the molten state may be the reason for the high value of tan δ for more clay amount composites.[147] The loss factor increases with the increase in the concentration of clay which is mainly due to the interaction of polymer and clay. Concisely the rheological report indicates that there is a stronger interaction between kaolin clay and terpolymer chains. Frequency sweep experiments were used to investigate the melting behavior of the terpolymer/clay composite samples. The plot of complex modulus (G*) on frequency is shown in Figure 3.8. Throughout the frequency range, the modulus decreases as the frequency increases.

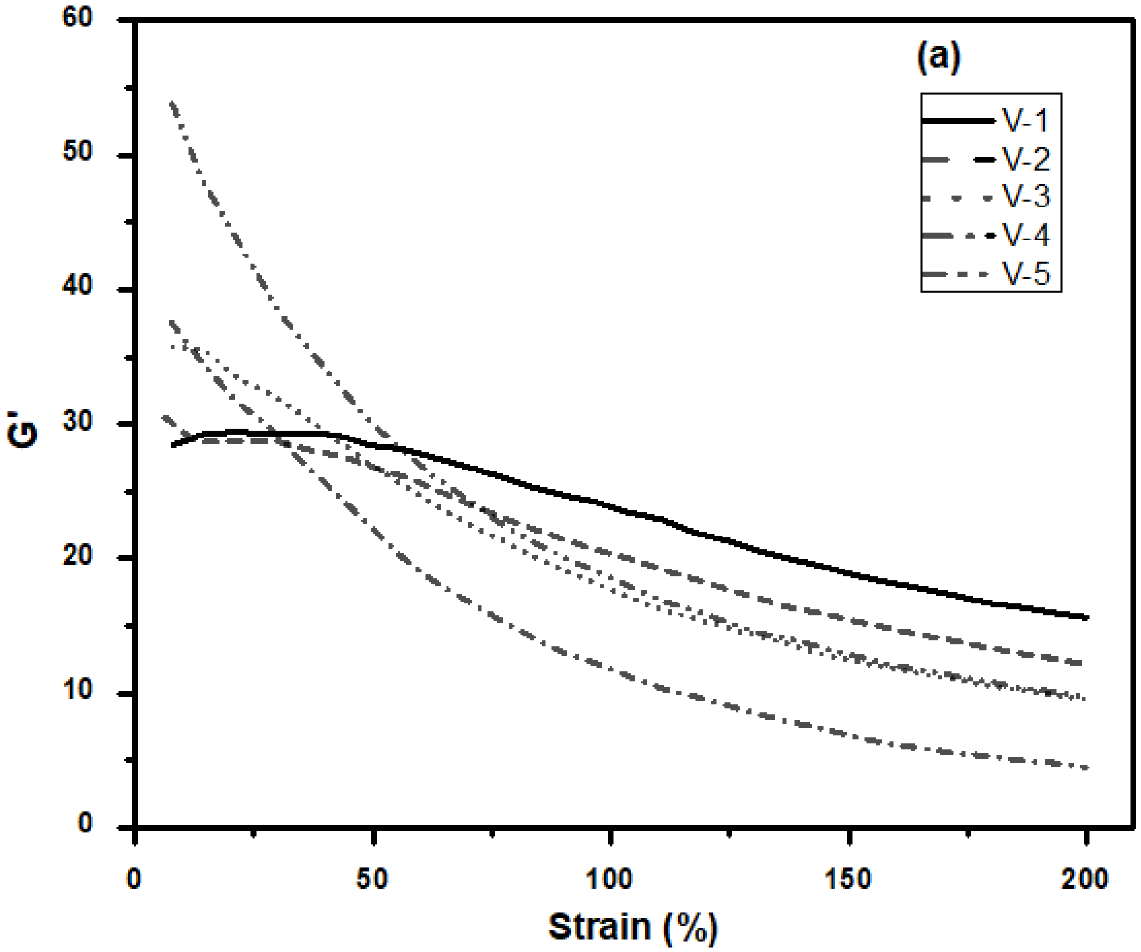


Figure 4.7. (a) Storage modulus vs strain % plots for the terpolymer and its composites.

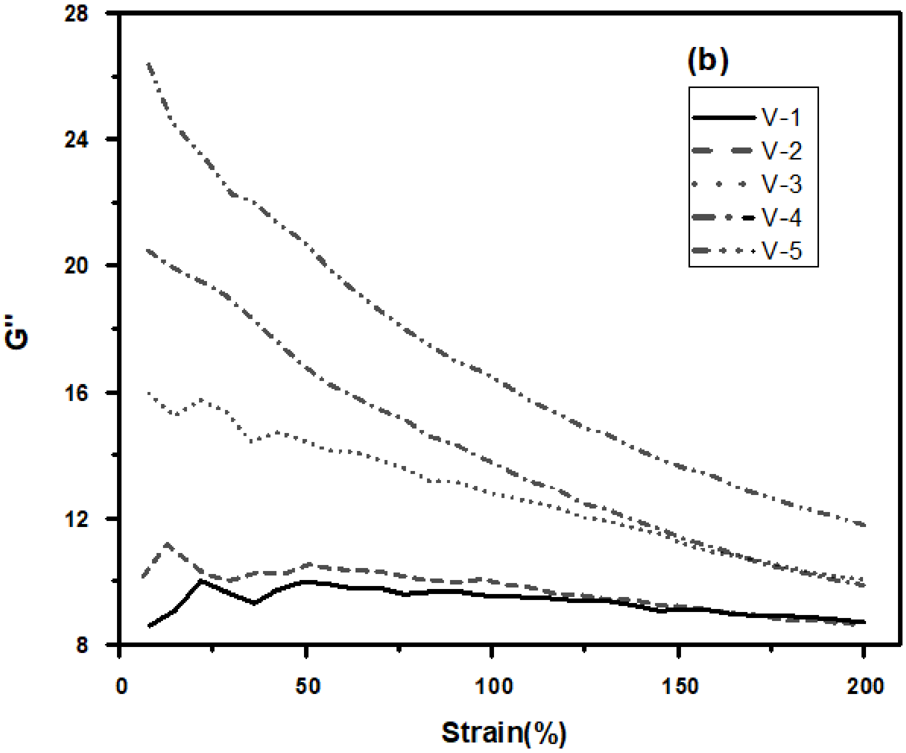


Figure 4.7.(b) Loss modulus plots for the terpolymer and its composites.

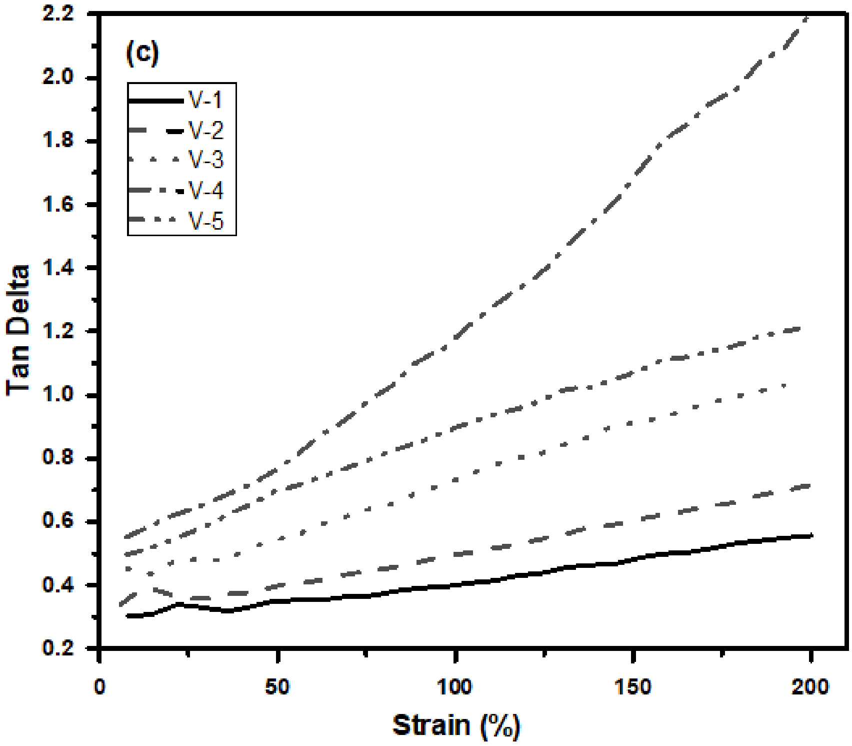


Figure 4.7.(c) Loss tangent plots for the terpolymer and its composites.

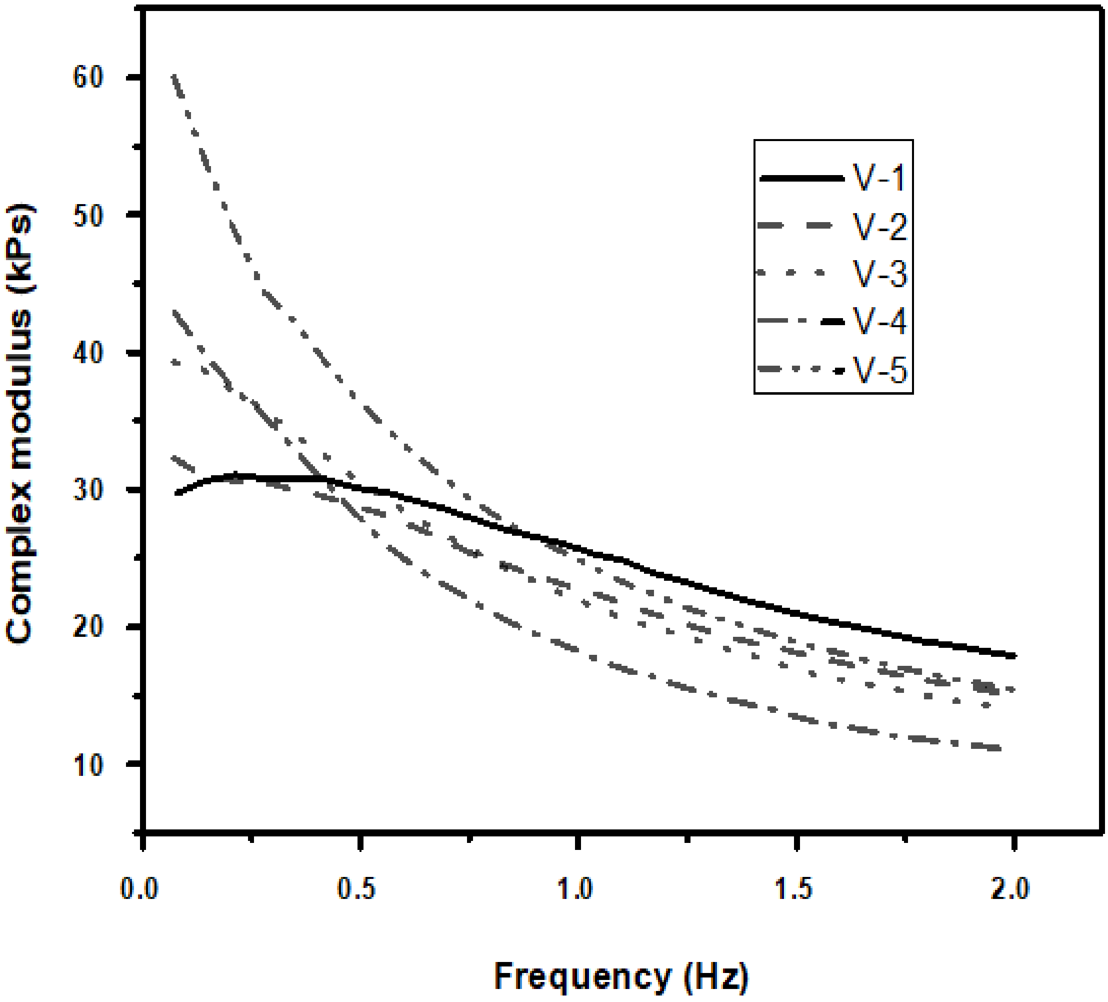


Figure 4.8. Complex modulus as a function of frequency (Hz) of terpolymer and terpolymer/clay composites.

Figure 4.9.(a) depicts the relationship between storage viscosity (dynamic viscosity) and frequency. The results show that storage viscosity (η') decreases as the frequency of storage increases. According to Datta *et al.*, the real component of the complex viscosity ($\eta^* = \eta' - i\eta''$) is called the storage viscosity for the melt as a fluid in the theory of viscoelasticity. The loss modulus G″ is represented by ($\eta' = G''/\omega$), whereas η″ is the imaginary component representing the melt elasticity, and the relation between η″ and the storage modulus G″ is $\eta'' = G'/\omega$.[145]

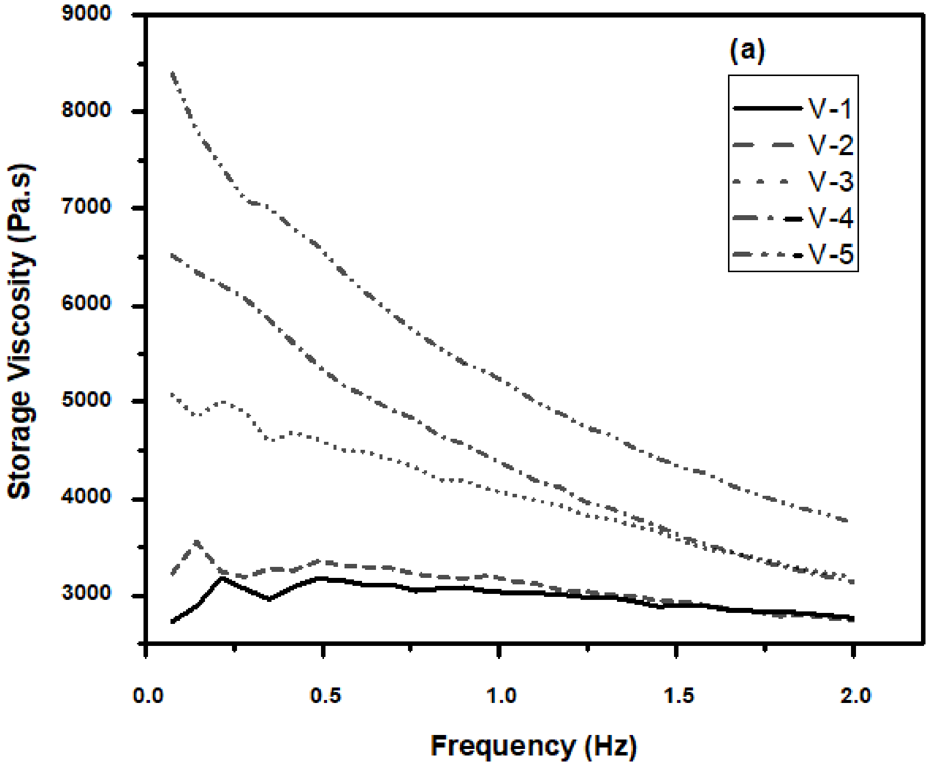


Figure 4.9.(a) Storage viscosity of terpolymer and terpolymer/clay composites.

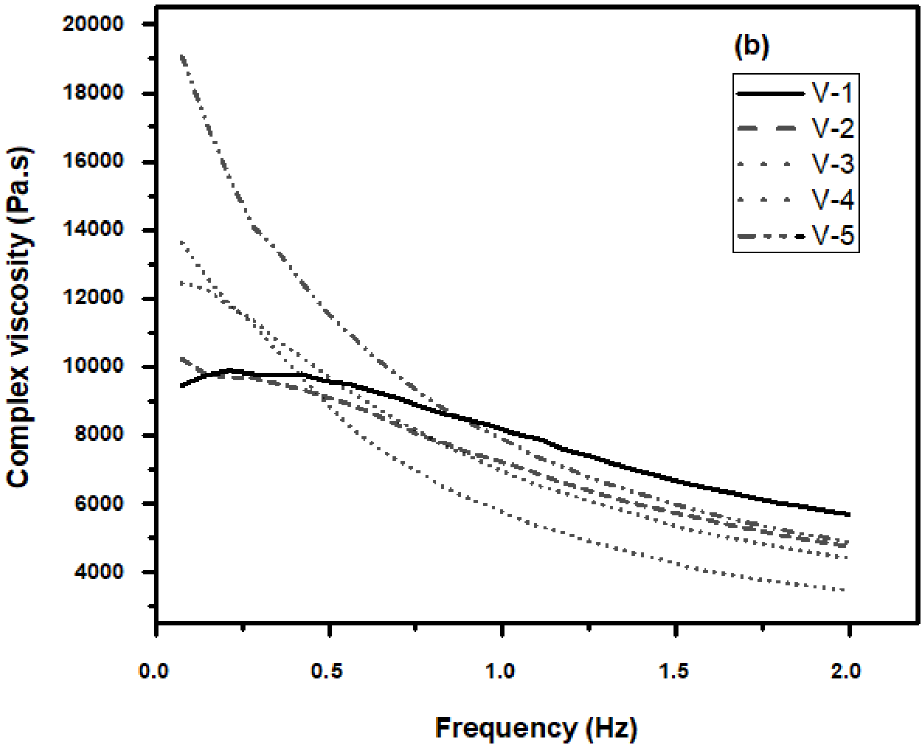


Figure 4.9.(b) Complex viscosity of terpolymer and terpolymer/clay composites.

In frequency sweep mode, the complex modulus of sample V-1 was higher than that of sample terpolymer/Clay composites V-2. It is presented that the incorporation of clay eagerly decreases the modulus from 27.08 kPa for sample V-1 to 24.4 kPa for sample V-2 at a frequency of 83.17 %. In the comparison of the outcomes of the complex modulus for terpolymer and terpolymer/clay composites samples with increasing the clay concentration indications that terpolymer/clay composites have a more pronounced filler effect using clay of its low molecular weight and therefore the low quantity of melt viscosity. This performance and amount of modulus development at the low-frequency region may be due to higher interaction of clay and polymer chains for the reason of more effective clay dispersion.[148] Figure 4.9.(b) shows the plot of complex viscosity as a function of frequency for polymer/clay composites. The results showed that as the shear rate or frequency range increases, the complex viscosity (η^*) of all samples decreases. This is due to the fact that all polymer composites exhibit pseudoplastic or non-Newtonian fluid behavior. As a result, the polymer composites have good processing characteristics.[147] The storage viscosity increases with an increase in the clay amount. The complex viscosity decreases with the increase in clay amount. These rheological responses indicate a stronger interaction between kaolin clay and terpolymer chains.

4.3.8. Morphological study

FESEM was used to examine the surface morphologies of the polymer and the polymer/clay composites. The micrographs for polymer V-1 and polymer/clay V-2 composites are shown in Figure 4.10. According to these micrographs, polymer composites have a smaller particle size than that of polymer/clay composites. When the clay is added, the particle size grows and agglomeration occurs.[149] This led to change the particle size in the terpolymer.

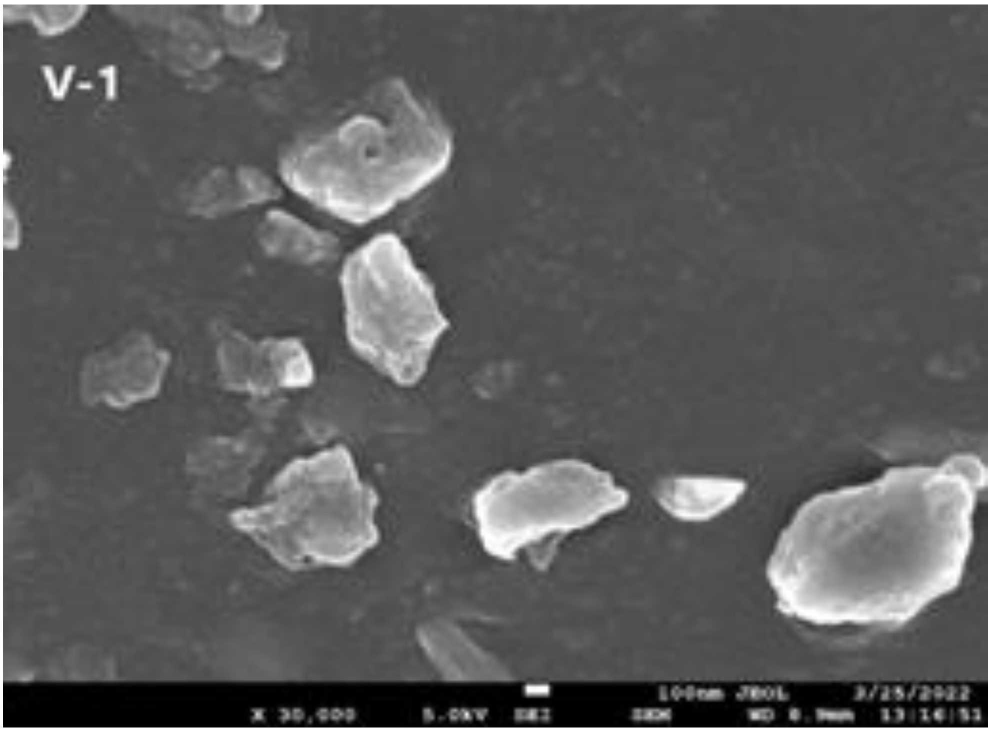


Figure 4.10. FESEM images of terpolymer (V-1).

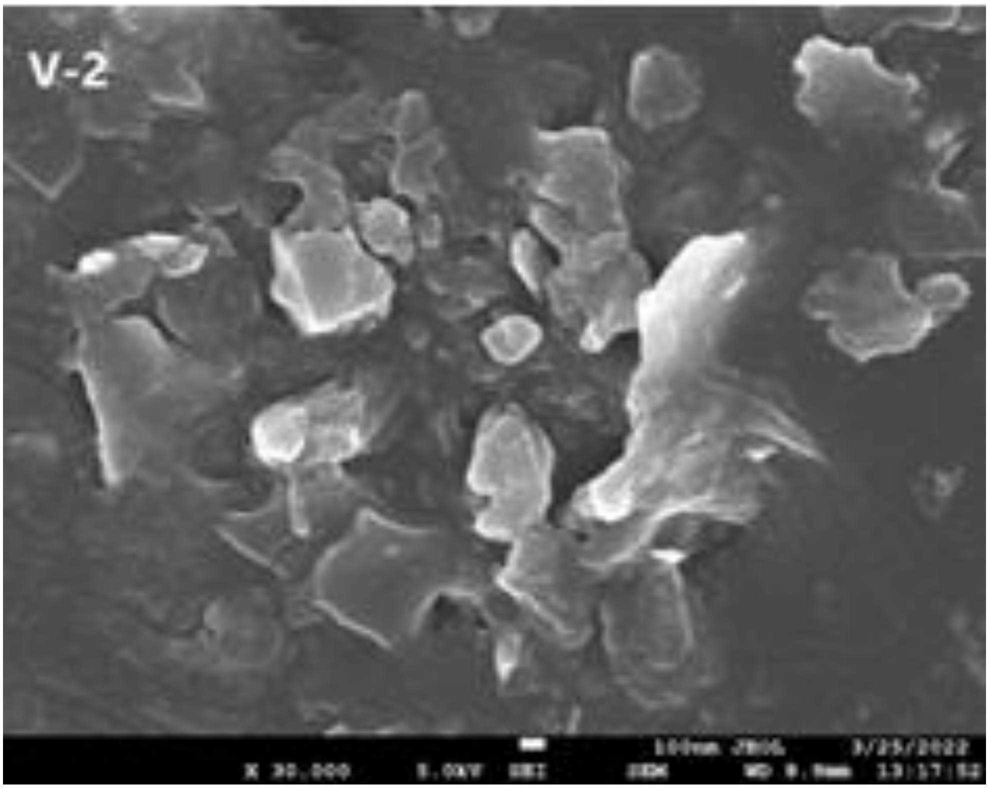


Figure 4.10. FESEM images of terpolymer/clay composite (V-2).

4.4. CONCLUSION

Conventional free-radical emulsion polymerization of VAc, BA, and AA yields PVAc-based terpolymer latex. The emulsion polymerization was successfully carried out at 70ºC using KPS as an initiator and SDS as a surfactant. The conversion of the monomers after one hour of polymerization was calculated gravimetrically and found to be 90%. The successful incorporation of co-monomers in the terpolymer was confirmed by ^{1}H NMR analysis. A requisite amount of clay is mixed directly with the terpolymer latex to prepare the composites. FT-IR studies reveal a distinct interaction between the >Al=O groups of the clay with that of the carbonyl group in the polymer. The bonding of clay to the polymer chain was confirmed by DSC analysis, where an increase in T_g of the composites was observed with clay loading. According to the TGA and DTG analyses, degradation temperature rises as clay content increases indicating that clay increases the thermal stability of the composites. As a result, the thermal properties of the composites have improved. The interaction between the polymer and clay was also confirmed via tensile measurements. The tensile strength decreased as the percentage of kaolin composites increased. With the increase of filler loading the uniform dispersion and adhesion of clay into the polymer matrix increases and that increases the tensile modulus. As a result, clay-based composites improve the physicomechanical properties of polymer films. It was observed that the presence of clay enhances the viscous properties of the polymer composites in the molten state using an RPA in strain sweep mode. Experiments with frequency sweeps show that the complex viscosity of all composites decreases as the frequency range increases. As a result, polymer composites exhibit pseudoplastic or non-Newtonian fluid behavior. Thus, it can be concluded that the polymer composites have good processing characteristics and may have several potential applications as adhesives.