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Simultaneous Improvement of Mechanical Properties and Thermal Stability of Polyvinyl Chloride Nanocomposite by Magnesium Aluminum-Layered Double Hydroxide

Ehsan Asghari[1], Alireza Azizi[2,*]

Abstract

The application of nanoclays in polyvinyl chloride (PVC) is limited as the organic modifier of conventional nanoclays can degrade PVC. Using usual nanoclays could reduce thermal stability due to the catalytic effects on PVC degradation, so the main chalenge is to find suitable nanoclay for PVC. The effects of two nanoclays, MgAl-layered double hydroxide and alkylammonium-modified montmorillonite, were studied on the mechanical properties of PVC nanocomposites. Dioctyl phthalate (DOP)-plasticized PVC was used as a matrix. Samples containing 1 phr, 3 phr and 5 (phr) of nanoclay were prepared and their mechanical and rheological properties were measured. Alkylammonium-modified montmorillonite nanoclay decreased the thermal stability of PVC despite the improvements in mechanical properties. This type of nanoclay decreased the thermal stability, causing a color change from yellow to pink with increasing the nanoclay content. Simultaneous improvement of mechanical properties and thermal stability of PVC nanocomposites were observed upon using MgAl-layered double hydroxide nanoclay. The results of the dynamic mechanical thermal analysis (DMTA) test showed an increment in the storage modulus and glass transition temperature with the use of MgAl-layered double hydroxide nanoclay, suggesting the significant effect of this type of nanoclay. The results of rheometer mechanical spectroscopy (RMS), however, showed no significant difference in the complex modulus viscosity and loss modulus.

Graphical abstract: *The effect of two types of Magnesium Aluminum-layered double hydroxide (MgAl-LDH) nanoclay and alkylammonium-modified montmorillonite (OMMT) was investigated on the and thermal stability of polyvinyl chloride (PVC) composite. The addition of MgAl-LDH nanoclay increased the storage modulus and glass transition temperature. In addition to improving mechanical properties, thermal stability was also enhanced.*

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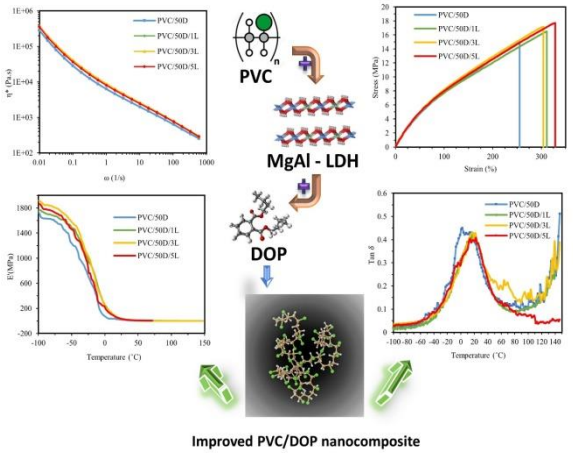

Keywords: Nanocomposite, Nanoclay, PVC, Mechanical properties, Thermal stability.

INTRODUCTION

Polyvinyl chloride (PVC) enjoys unique properties such as good mechanical properties, high chemical stability and good electrical insulation properties. PVC degrades upon exposure to heat, oxygen, and sunlight due to the formation of unsaturated polymer chains. With the loss of hydrochloric acid and degradation of polymer chains, color of PVC changes to yellow or brown. Thermal stability is usually improved by adding thermal stabilizers or nanoclays. For this reason, PVC is usually alloyed with special additives or other polymers to achieve better mechanical and thermal properties [1-3].

Nanocomposites can cover desired features by taking the advantage of the characteristics of several materials at the same time. Polymer nanocomposites have been widely used in recent years. Nanoparticles can resolve the problems of PVC products through different mechanisms. In general, proper incorporation of the second phase into the polymer matrix can significantly improve the properties of the final product [4-8].

In 2004, Wan et al. investigated the effects of quaternary alkylammonium on the thermal stability of montmorillonite composites modified with quaternary alkylammonium PVC/OMMT. PVC-sodium montmorillonite composites were also prepared. PVC/OMMT composites showed better mechanical properties and higher thermal stability than PVC/MMT composites. However, under the same melt process conditions, color of PVC/OMMT composites changed from light yellow to pink with increasing OMMT content, while color of PVC/MMT composites did not change. Degradation of the alkylammonium modifier and catalytic effects on PVC dechlorination are the main factors in the discoloration of PVC/OMMT composites [9].

In another study in 2012, Saad et al. mixed PVC and DOP with 5 and 10 wt.% of Cloisite Na+, Cloisite 30B and Cloisite 93A. The nanocomposites were characterized by TGA, which showed that thermal stability rose in the following order: Cloisite Na+ < Cloisite 93A < Cloisite 30B [10].

In 2011, Liu et al. investigated the enhancement of mechanical properties of PVC combined with halloysite nanoclay (HNT) cross-linked with polymethyl methacrylate (PMMA) [11]. The results showed that HNTs grafted with PMMA can effectively improve the toughness, strength, and modulus of PVC. The glass transition temperature (T_g) of the PVC phase in PVC/PMMA-grafted HNTs nanocomposites also shifted to slightly higher temperatures.

Nanoclays could improve the mechanical properties of PVC. These nanostructures serve as reinforcing agents within the PVC matrix, incrementing the tensile strength, elastic modulus, and flexibility. The presence of nanoclay ensures a more uniform distribution of the polymer structure, preventing the formation of weak regions within the material. Additionally, nanoclays can inhibit the propagation of cracks and prevent sudden failures, thereby, increasing the impact resistance of the polymer [14].

To achieve the best results, nanoclays must be uniformly dispersed within the PVC matrix. This is typically achieved through extrusion or melt blending. A significant improvement can be achieved in the thermal and mechanical properties of PVC by optimizing the nanoclay content and its dispersion while maintaining other properties of the polymer. In conclusion, the incorporation of nanoclay could be an effective and efficient method to overcome the limitations of PVC, making it more suitable for demanding industrial and engineering applications [14].

The incorporation of nanoclay into PVC can enhance its thermal stability. Nanoclays act as physical barriers, preventing heat transfer and delaying the decomposition of the polymer. Consequently, the rate of hydrogen chloride (HCl) release is reduced, and the thermal degradation of PVC is postponed. Furthermore, upon uniform dispersion within the PVC matrix, nanoclays can serve

as diffusion barriers for the gases generated during degradation, thereby, increasing the thermal stability. Ultimately, by raising the decomposition temperature and reducing the degradation rate, PVC can be used at higher temperatures with no need for traditional thermal stabilizers [3]. But there is a problem! Using usual nanoclays could reduce thermal stability due to the catalytic effects on PVC degradation, so the main chalenge is to find suitable nanoclay for PVC.

The main purpose of this research is to find suitable nanoclay for plasticized PVC capable of simultaneous improvement of the mechanical properties and thermal stability of PVC. Tow type of nanoclay were used and results were discussed.

EXPERIMENTAL

Materials and Devices

Polyvinyl chloride (PVC) with K-Value of 65 was obtained from Ghadir Petrochemical Company of Iran. Dioctyl phthalate (DOP) plasticizer with a density of 0.985 g/cc was provided from Rama Fidar Rad Chemical Company, OMMT nanoclay with a 3.6 nm plate spacing was obtained from Zhejiang Fenghong Chemical Company, China. MgAl Layered double hydroxide nanoclay with a 1.5 nm plate spacing was purchased from Nano Parmin Khavaran Chemical Company, Iran, while lead-based thermal stabilizer was obtained from Hampar Company. The magnetic stirrer of Sahand Chemical Company was used for the initial mixing of the materials, Figure 1. The mechanical stirrer was used for mixing the materials, Figure 2. The Rheosense internal mixer manufactured by the Iranian Polymer and Petrochemical Research Institute was utilized to prepare the samples, Figure 3. Hot Press machine was used for samples preparation, Figure 4.

STM-20 stretching device manufactured by Santam Company was applied for tensile testing at the test speed of 50 mm/min, Figure 5. The samples were prepared according to ASTM D638. Dynamic mechanical thermal analysis was performed from -100 °C to 150 °C under a frequency of 1 Hz and the heating rate of 10 °C/min by a DMTA-TRITON TRITEC 2000 DMA STARE SYSTEM model. The dynamic shear properties were measured using the eccentric rotatory disc (ERD) mode of the Rheometrics mechanical spectrometer. The measurements were made over the frequency region of 0.01 to 600 radians per sec at a temperature of 175°C. The dynamic strain was one percent. The thickness and diameter of the samples were kept the same in all cases.

The thermal stability of the samples was evaluated in accordance with the national standard of Iran No. INSO 5525-405, 2020 edition (IEC 60811-405). To measure and compare the degradation time of the samples, a part of the prepared film with approximate dimensions of 1x1 cm^2 was separated. Then,

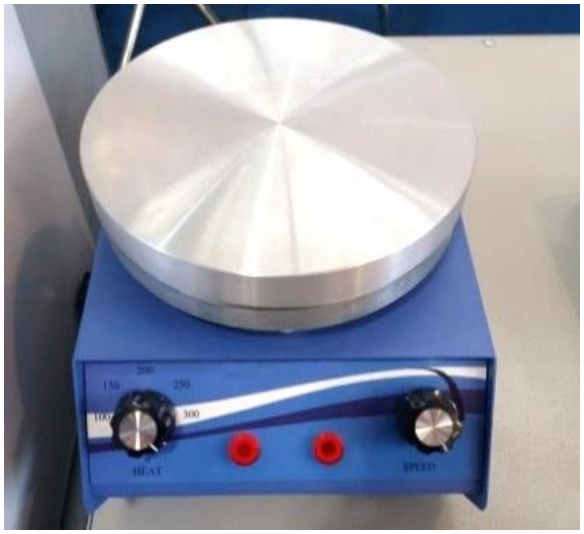

Figure 1. Magnetic stirrer

Figure 2. Mechanical stirrer

Figure 3. Internal mixer

Figure 4. Hot Press machine

Figure 5. Tensile test machine

Figure 6. Thermal stability test machine

it was cut into small pieces and poured into the capillary tube, Figure 6. pH paper was placed on top of the capillary tube and was heated to 200ºC . After observing the color change in the paper, the time was recorded. The recorded time is the degradation time of the PVC or the time the polymer can withstand the heat. For each sample, this test was conducted three times.

Methods

Nanoclay and dioctyl phthalate were first mixed with a magnetic stirrer for 5 minutes. For better mixing, the weighed samples were mixed by a mechanical mixer for 10 minutes at 1000 rpm before being added to the internal mixer.

Nanocomposites were prepared by melt mixing PVC, nanoclay, and various processing additives in the chamber of internal mixer. The mixing temperature was set at 170 °C, the rotor speed was 50 rpm, and the mixing time was 4 min. The prepared nanocomposites were then molded into sheets of 2 mm in thickness by hot pressing at 185 °C and 20 MPa for 7 min, followed by cooling to room temperature at 5 MPa. Next, the prepared samples were tested, Table 1. The experimental samples shown in Figure 7.

Table 1. Composition of samples (phr).

Samples	PVC (phr)	DOP (phr)	OMMT nanoclay (phr)	Mg-Al-LDH nanoclay (phr)	Stabilizer (phr)
PVC/50D	100	50	0	0	5
PVC/50D/1K	100	50	1	0	5
PVC/50D/3K	100	50	3	0	5
PVC/50D/5K	100	50	5	0	5
PVC/50D/1L	100	50	0	1	5
PVC/50D/3L	100	50	0	3	5
PVC/50D/5L	100	50	0	5	5

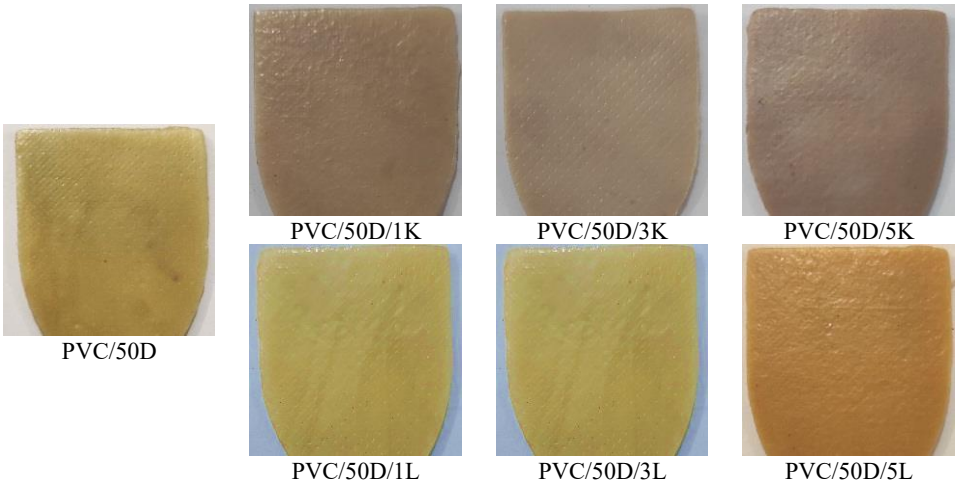

Figure 7. Experimental samples.

RESULTS AND DISCUSSION

Tensile Test

Effect of OMMT Nanoclay on PVC/DOP Composite

The results of mechanical tests were performed on PVC/OMMT nanocomposites with OMMTs loadings varying from 0 phr to 5 phr, are listed in Table 2. The tensile strength and elongation at break of the three PVC/OMMT nanocomposites were improved at OMMT loadings of 1 phr to 5 phr. Figure 8 shows the stress-strain curve. As seen, the addition of OMMT nanoparticles to the PVC/DOP blend increased tensile strength and elongation at break at all contents.

The tensile strength decreased by raising the OMMT nanoclay content due to clumping in the polymer matrix, which decreased the tensile strength, fracture energy, and modulus. The increase of nanoclay content also led to clumping of nanoclay which could act as points of stress concentration and crack initiation, resulting in premature failure of the sample. The same results of nanoclay agglomeration have also been reported in other studies and its negative effects on tensile properties were also observed at high nanoclay contents [12-14]. As can be seen, the modulus and fracture energy also decreased, Figure 9.

Table 2. Mechanical properties of PVC/OMMT nanocomposites.

Samples	OMMT content (phr)	Tensile strength (MPa)	Elongation at break (%)	Young modulus (MPa)	Fracture Energy (J)
PVC/50D	0	15.16±0.53	291±32.35	8.8±0.17	11500±830
PVC/50D/1K	1	18.14±0.56	347±37.29	8±0.19	9310±1990
PVC/50D/3K	3	16.41±0.91	343±23.8	8.33±0.22	8836±700
PVC/50D/5K	5	17.55±0.67	335±5.69	11.3±0.35	9409±260

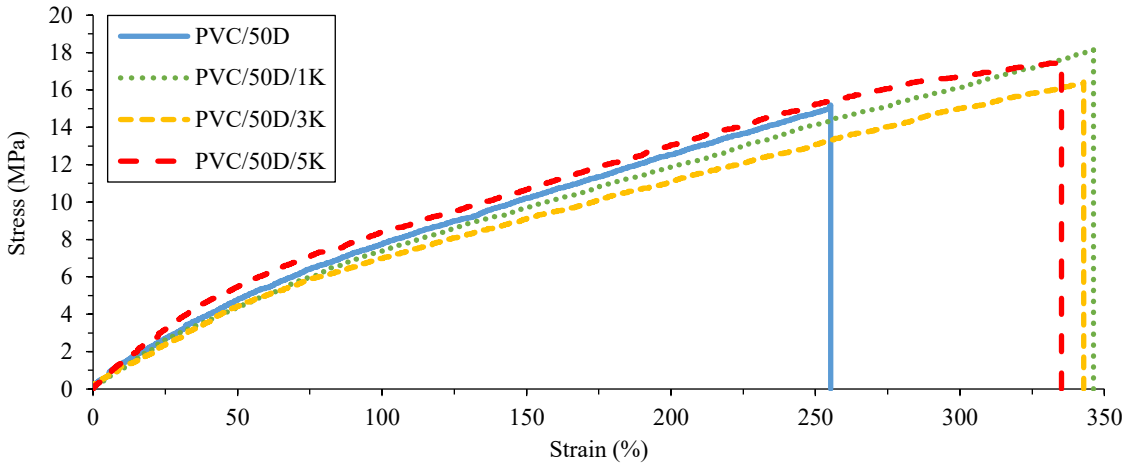

Figure 8. Tensile Stress-Strain curve of PVC and PVC/OMMT nanocomposites.

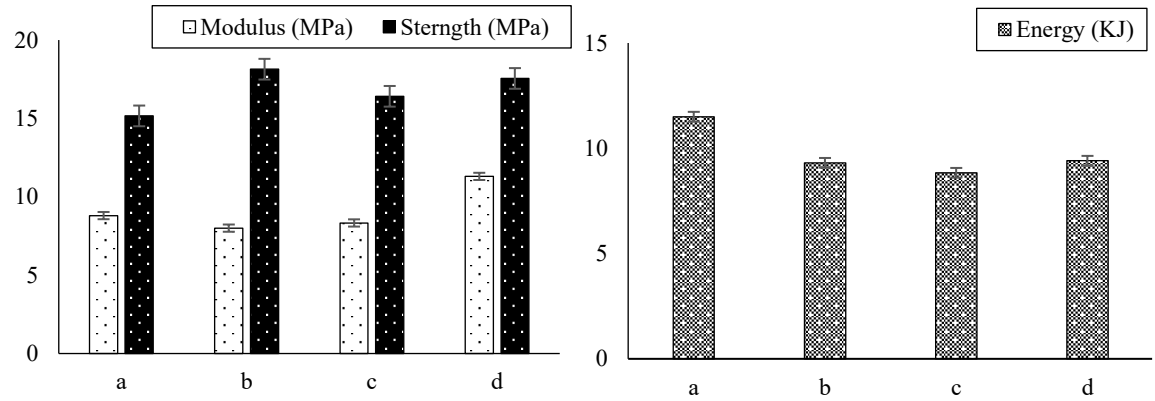

Figure 9. The effect of OMMT nanoclay on the mechanical properties of PVC for (a) Without nanoclay (b) 1 phr OMMT; (c) 3 phr OMMT; (d) 5 phr OMMT; plasticized with DOP

Effect of LDH Nanoclay on PVC/DOP Composite

The results of mechanical tests were performed on PVC/LDH nanocomposites with LDHs loadings varying from 0 phr to 5 phr, are listed in Table 3**.** The mechanical properties of the three PVC/LDH nanocomposites were improved at LDHs loadings of 1 to 5 phr. The addition of LDHs nanoparticles to the PVC/DOP blend increased tensile strength at all contents (see Figures 10 and 11).

Tensile strength depends on the dispersion of nanoclays and their adhesion the mixture substrate [11]. The incorporation of nanoparticles into the polymer and their dispersion in the polymer matrix promote adhesion between the polymer and the nanoparticles, therefore, increasing tensile strength. Thus, tensile strength of the nanoparticle-containing polymer is higher than that of the pristine polymer (without nanoparticles). A comparison of tensile strength of the LDH-containing samples indicates the much higher tensile strength of the PVC/D50/5L sample due to the higher intercalation of the nanolayers and their more uniform distribution in the sample.

Table 3. Mechanical properties of PVC/LDH nanocomposites.

Samples	LDH content (phr)	Tensile strength (MPa)	Elongation at break (%)	Young modulus (MPa)	Fracture energy (J)
PVC/50D	0	15.16 ± 0.53	291 ± 32.35	8.8 ± 0.17	11500 ± 830
PVC/50D/1L	1	16.51 ± 0.09	311 ± 2.65	8.12 ± 0.10	9893 ± 210
PVC/50D/3L	3	17.16 ± 0.62	304 ± 16.09	9.36 ± 0.18	10182 ± 1070
PVC/50D/5L	5	17.72 ± 0.43	328 ± 9.24	9.66 ± 0.17	11300 ± 400

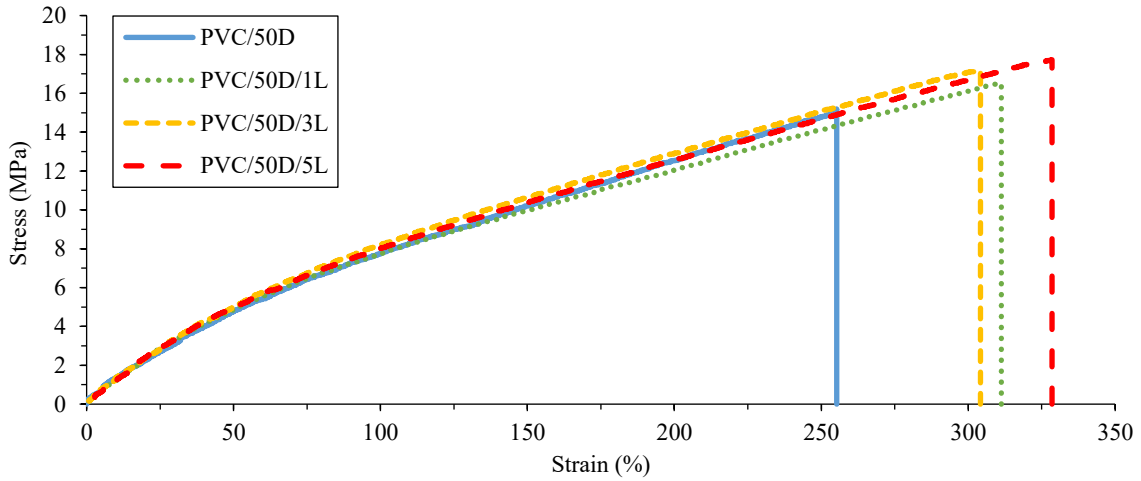

Figure 10. Stress curve according to the strain of the samples for PVC and PVC/LDH nanocomposites.

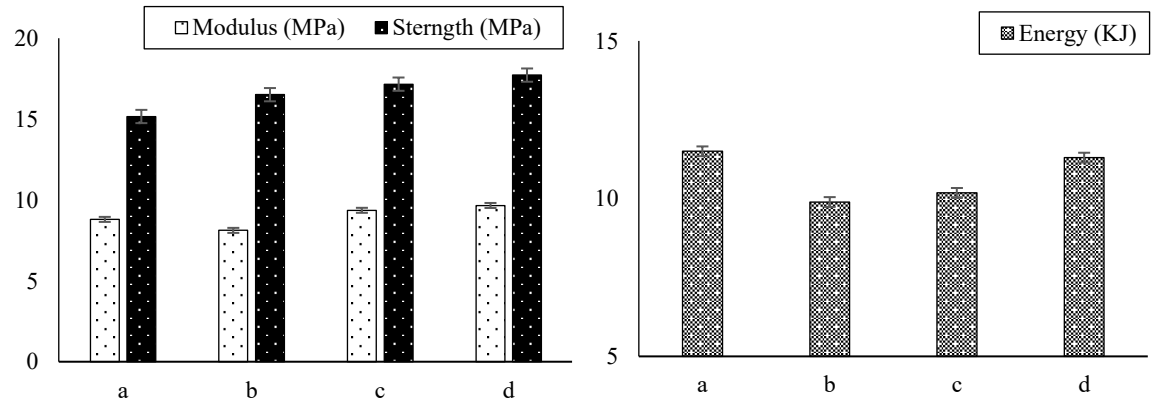

Figure 11. The effect of LDH nanoclay on the mechanical properties of PVC for (a) Without nanoclay (b) 1 phr LDH; (c) 3 phr LDH; (d) 5 phr LDH; plasticized with DOP

According to previous studies [15], an increase in nanoclay content up to 5 phr in PVC-nanoclay nanocomposites can improve the mechanical properties. In this study, nanocomposites containing 3 phr to 5 phr of nanoclay had the greatest effect on mechanical properties. These results are also observed in the mechanical properties obtained from the tensile test.

The Young's modulus of the LDH-containing blends increased by elevating the nanoparticle content. The reason can be attributed to the increase in interfacial adhesion between the dispersed LDHs nanolayers and the PVC matrix. These samples also exhibited higher adhesion and nanoparticles-polymer matrix contact area compared to the pristine sample, which restricts chain orientation and increases the modulus. The fracture energy has gradually decreased, indicating a decline in the toughness of the samples. This indicates that the addition of nanoparticles has reduced toughness.

Thermal Stability

Autocatalytic dehydrochlorination is known as the main process in thermal degradation of PVC. Table 4 shows the results of the thermal stability tests. The thermal stability of samples was measured and based on the type and amount of nanoclay significant differences were observed. Thermal stability of PVC was significantly reduced by the addition of OMMT nanoclay. The PVC/50D/1K sample showed a thermal stability of 12 minutes, which decreased further to 8 minutes in PVC/50D/3K and 4 minutes in PVC/50D/5K. In contrast, by the addition of MgAl-LDH nanoclay thermal stability was improved. The PVC/50D/1L sample was demonstrated the highest thermal stability of 65 minutes.

Table 4. The thermal stability of PVC and PVC nanocomposites

Samples	Thermal stability (min)
PVC/50D	57±1
PVC/50D/1K	12±1.2
PVC/50D/3K	8±0.9
PVC/50D/5K	4±1.1
PVC/50D/1L	65±0.8
PVC/50D/3L	62±0.9
PVC/50D/5L	60±1

According to previous works on the effects of montmorillonite nanoclay modified with type IV alkyl ammonium on thermal stability, this nanoclay decreased the thermal stability, causing a color change from yellow to pink with increasing the nanoclay content. The decomposition of the fourth type alkyl ammonium modifier and the catalytic effects on PVC dechlorination are the main reasons for color change and reduced stability [9].

A catalytic effect was observed on the dechlorination of PVC in the samples containing OMMT nanoclay, which caused a severe decrease in thermal stability. As the content of OMMT nanoclay increased, the thermal stability decreased due to the enhanced catalytic activity of the nanoclay particles, which accelerated the dechlorination process. This effect can be attributed to the increased surface area and reactivity of the nanoclay, which promotes the degradation of PVC at lower temperatures. Additionally, excessive loading of the nanoclay can lead to the formation of weak sites within the polymer matrix, further contributing to the reduction in thermal stability [16].

It is well seen that layered double hydroxide are good thermal stabilizers for PVC, therefore, PVC/LDH nanocomposites showed longer stability (above 60 min). According to Table 4, the stabilization time of PVC/LDH nanocomposites was higher than PVC compounded solely with the DOP plastisizer and lead heat stabilizer.

In the samples that used LDH nanoclay, the thermal stability not only did not decrease but also showed a favorable increase as this nanoclay is a layered double hydroxide nanoclay. The increase in the thermal stability can be attributed to the electrostatic interaction between the electron cloud of chlorine atoms in the PVC chain and the positive charge of the LDH layer. Such an interaction reduces the electron cloud density of chlorine atoms, thereby, weakening its activity and hindering the initiation of the chlorine reaction [17].

The stabilization activity can be ascribed to the capacity of the LDHs to react with the HCl formed during PVC degradation. This reaction occurs in a two-step process: if possible., the counterions first of the LDH-layers tend to react with the HCl gas, then, LDHs themselves react with the HCl with complete destruction of the LDH structure and formation of metal chlorides. By delaying the reaction of chlorine with the PVC chain, the degradation of this polymer could be also delayed which increases the thermal stability [18]. According to the thermal stability results, the samples containing OMMT nanoclay were analyzed.

Dynamic Mechanical Thermal Analysis (DMTA)

Storage modulus, glass transition temperature, and full width at half maximum (FWHM) were obtained from dynamic mechanical thermal analysis test, Table 5. Figure 12 shows the dynamic mechanical spectra of storage modulus (E') as a function of temperature. As seen, below glass transition region, the storage moduli (E') of the three different PVC/LDH nanocomposite samples are all higher than that of pristine PVC. In addition, E' of PVC/50D/3L composite is slightly higher than those of the PVC/50D/1L and PVC/50D/5L composites.

Table 5. Transition temperatures T_g and dynamic storage moduli of the PVC/LDH composites

Samples	Storage modulus, E′ (MPa)				Full width at half maximum, FWHM (°C)	Glass transition temperature, T_g (°C)
	-100 °C	*0 °C*	*50 °C*	*150 °C*		
PVC/50D	1720.57	98.87	5.46	0.13	50	2.3
PVC/50D/1L	1752.03	250.78	7.88	0.24	54	18.6
PVC/50D/3L	1914.10	219.02	3.68	0.14	70	21.5
PVC/50D/5L	1866.45	230.50	5.12	3.42	56	22.5

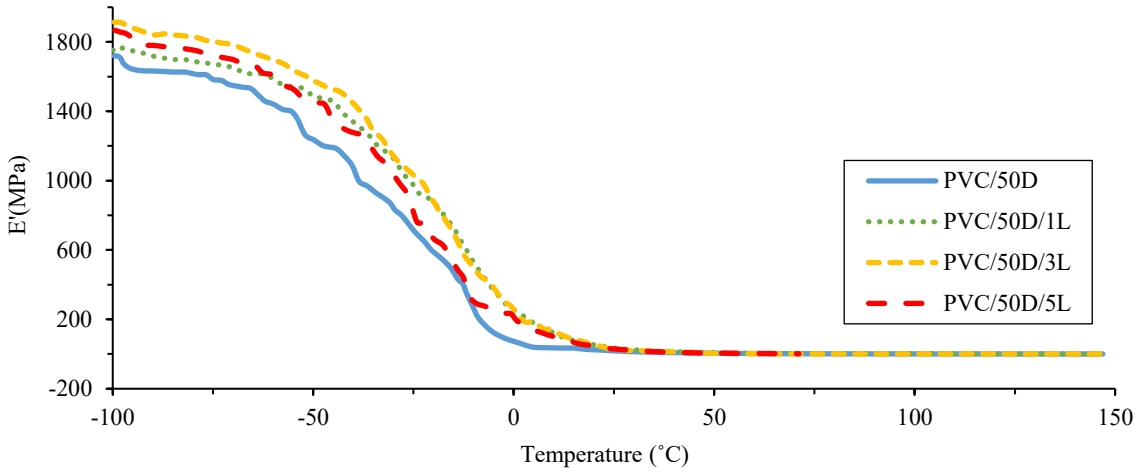

Figure 12. Temperature dependences of E' determined at 1 Hz for PVC and PVC/LDH nanocomposites.

The four materials showed no obvious differences in the rubber plateau modulus (E′). E' of the samples transferred to higher temperatures after the addition of nanoclay, suggesting an increment in the elasticity of the samples and the proper dispersion of nanoparticles.

The FWHM parameter slightly increased by adding nanoclay which was not significant. The broadening of the peaks depends on factors such as the size of the nanoclay particles, the presence of stress in the sample, and the error of the device. All possible factors are considered when interpreting the results [19].

Figure 13 displays the dynamic mechanical spectra of tan δ as a function of temperature for PVC/50D and PVC/LDH nanocomposites. One main transition (T_g) characteristic relaxation can be detected in the tan δ curves of nanocomposites within the tested temperature range. The transition temperature is determined from the peak temperatures in the curve of tan δ versus temperature.

As can be observed in Figure 13, between -100 and 150 °C the glass transition peaks of the three kinds of nanocomposite samples all shifted to higher temperatures compared to that of pristine PVC. The three different PVC/LDH nanocomposite samples showed similar values as their curves are matched.

The observed dynamic mechanical behavior can be explained as follows. First, the high surface area of nanoclay layers offers vast surface contact between PVC chains and nanoclay layers. Second, the radius of gyration of the polymer chain is much larger than the interlayer distance of the nanoclay, thus, the mobility environment of the polymer chains is limited when the polymer chains are interlayered [20].

The silicate layers of nanoclays limit the rotations of the PVC chain, which increases the storage modulus at low temperatures while raising glass transition temperature. Similar to the results of Wan et al., the nanoclay-containing samples were associated with an increase in E' and T_g compared to pristine PVC [21].

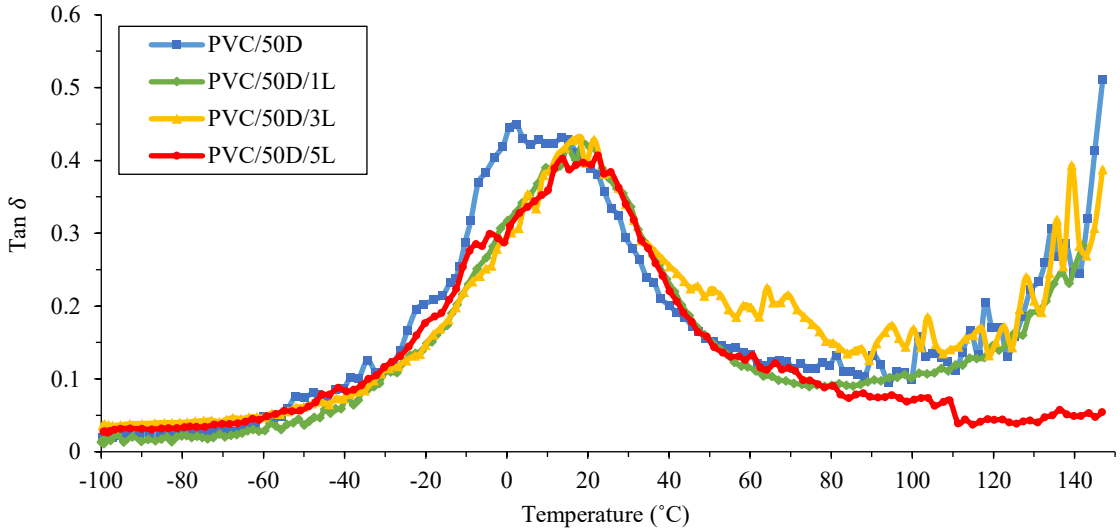

Figure 13. Temperature dependences of tan δ determined at 1 Hz for PVC and PVC/LDH nanocomposites.

As can be seen, the graph related to the PVC/50D/3L sample exhibits two separate peaks, suggesting a relaxation mechanism which occurs in two stages. This could be due to different energy barriers for the movement of PVC chains [22].

Rheometrics Mechanical Spectrometer (RMS)

Rheological tests are sensitive to molecule chain entanglements or chain structure such as small differences in chain length, branching, or networking. These factors will cause a large variation in flow behavior. A clear and accurate understanding of the rheological properties of various polymers or polymer composites is important in the polymer processing. Rheological behavior can provide information of the structure of materials and should be controlled for industrial applications

The complex viscosity of PVC/LDH system and PVC are presented in Figure 14. By addition of LDH to PVC, the complex viscosity increased, especially at low frequencies which can be attributed to the interaction of nanoclay in the polymer matrix. Furthermore, melt viscosity decreases with increasing frequency, exhibiting a pseudoplast behavior.

The dynamic storage modulus, G′, is related to the elastic behavior of material; while the dynamic loss modulus G″ represents the dissipated energy. The dependence of G′ and G″ on the frequency can offer a measure for the relative motion of all molecules in the bulk. The increase in elastic modulus G′ may result from the changes in molecular chain rigidity and the interaction between polymer chains.

Figures 15 and 16 show the logarithmic plots of G′ and G″ vs. frequency ω (Hz) for PVC/LDH nanocomposite, respectively. The G′ and G″ of nanocomposites exhibit a monotonic change with frequencies and in all samples are higher than PVC.

Larson RJ demonstrated that G′ and G″ are solid-like and liquid-like materials behavior, respectively. For the liquid-like behavior, the G′ is much lower than the G″, and vice versa for the solid-like system. In that case, the transition of melts from liquid-like to solid-like behavior occurs at crossover frequency (the frequency corresponding to the crosspoint of G′ and G″) [23]. In this work, the virgin PVC and the PVC/ LDH nanocomposite do not possess crossover frequency. For G′ > G″, the elastic response is similar to solid material at any frequency. Solid-like behavior has been observed in conventional filled polymer systems with strong interactions among the polymers and the fillers. The free rotation and movement of molecular chain was restricted, prevented from complete relaxation due to physical jamming. Thus, G′ and G″ increased upon adding LDH to PVC.

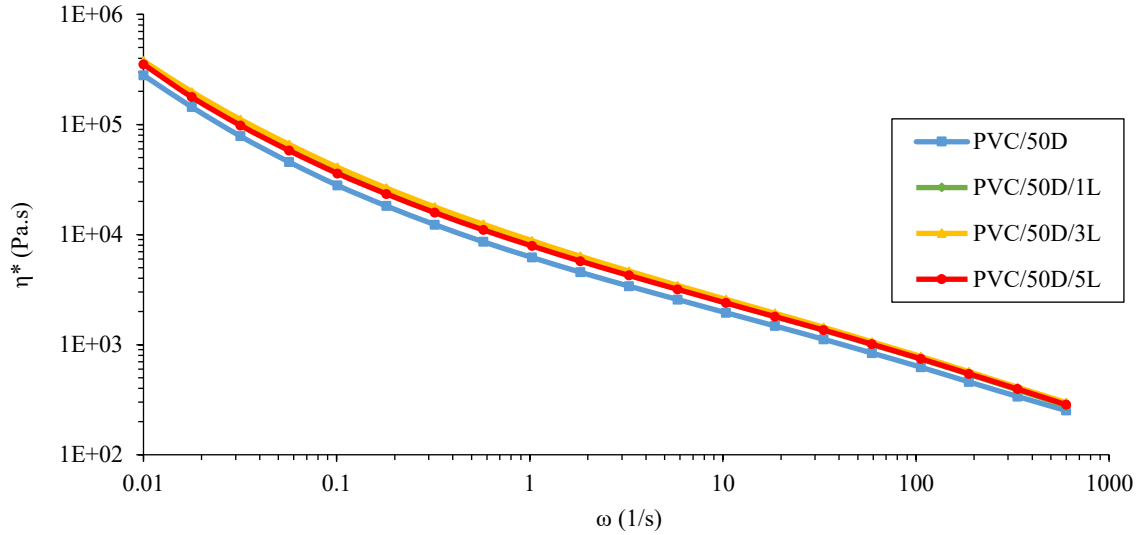

Figure 14. Complex viscosity of PVC and PVC/LDH nanocomposites

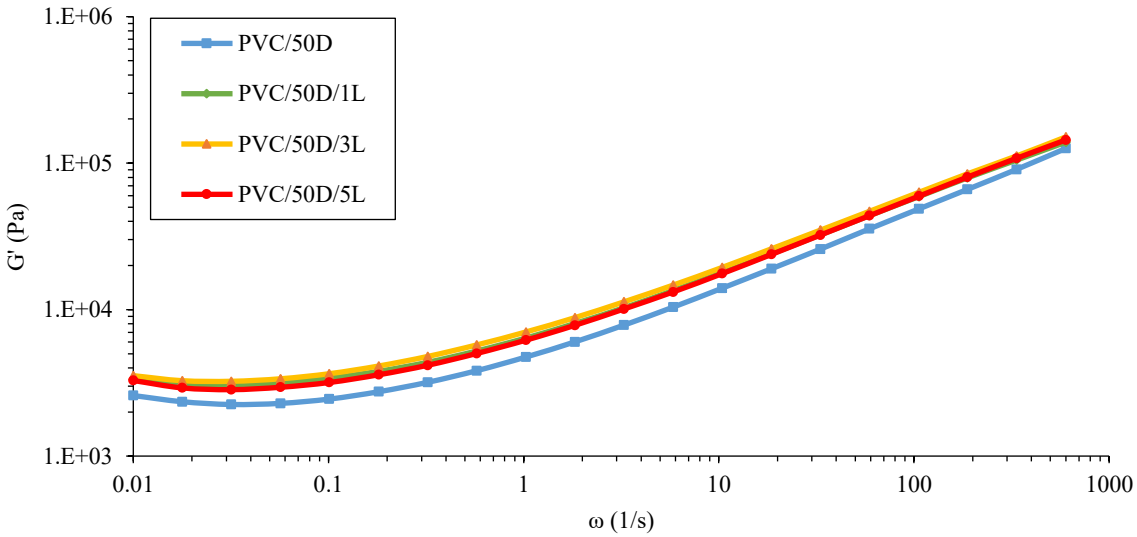

Figure 15. Storage modulus of PVC and PVC/LDH nanocomposites

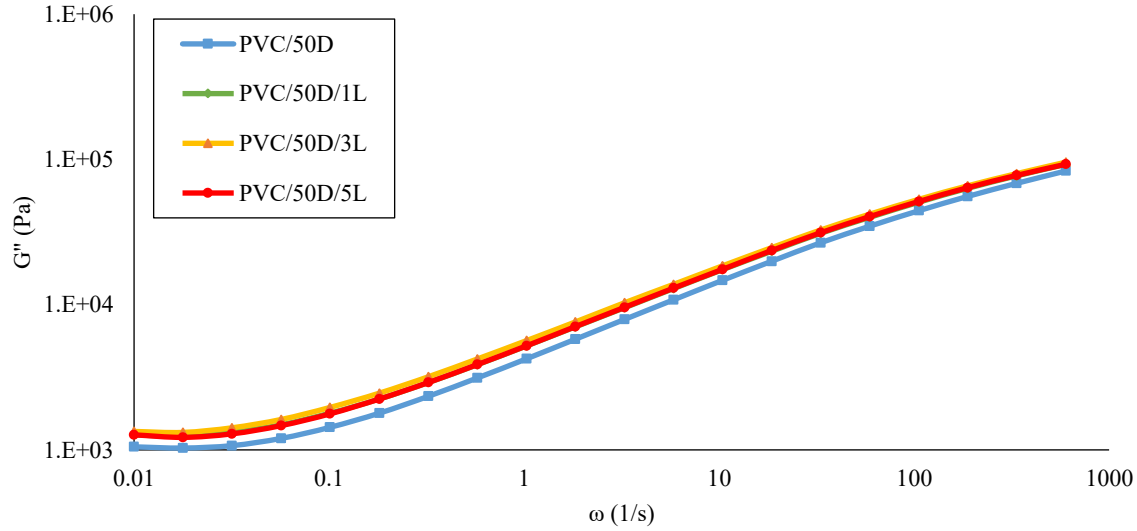

Figure 16. Loss modulus of PVC and PVC/LDH nanocomposites

CONCLUSION

In this research, layered double hydroxide nanoclay (LDH) and OMMT-modified montmorillonite were used to perpare PVC nanocomposites. The tensile test results showed that the addition of OMMT nanoclay to polyvinyl chloride increased the tensile strength, while declining the fracture energy and Young's modulus. By adding LDH nanoclay up to 5 phr, the tensile strength and Young's modulus increased, whereas the fracture energy showed no significant difference. Based on thermal stability results, the LDH-bearing samples showed more thermal stability compared to those lacking nanoclay and samples containing OMMT nanoclay. LDH nanoclay is a proper nanoparticle for polyvinyl chloride/dioctyl phthalate compounds as it prolongs thermal stability time and improves compatibility between nanocomposite components and nanoclay dispersion. The thermal stability of the samples containing OMMT nanoclay was severely decreased, thus, dynamic thermomechanical analysis and rheometric mechanical spectroscopy of these samples were not investigated. The dynamic thermomechanical test showed that the addition of LDH nanoclay elevated the storage modulus and the glass transition temperature was increased. The PVC/50D/3L sample exhibited the best uniformity and storage modulus. The results of the mechanical rheometric spectrometry test showed that the increase in the mixing viscosity of the samples containing LDH nanoclay is higher than the nanoclay-free sample. The increase in G' and G″ of the LDH-containing samples is higher than the sample without nanoclay at all frequencies, although it is not significant.

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