Structural Characterization and Thermal Properties of Chiral Poly(amide-imide)/Modified MgAl Layered Double Hydroxide Nanocomposites Prepared via Solution Intercalation

Shadpour Mallakpour, Mohammad Dinari

Organic Polymer Chemistry Research Laboratory, Department of Chemistry, Isfahan University of Technology, Isfahan, Islamic Republic of Iran

Nanotechnology and Advanced Materials Institute, Isfahan University of Technology, Isfahan, Islamic Republic of Iran

Accepted author version posted online: 29 May 2014. Published online: 20 Jun 2014.

To cite this article: Shadpour Mallakpour & Mohammad Dinari (2014) Structural Characterization and Thermal Properties of Chiral Poly(amide-imide)/Modified MgAl Layered Double Hydroxide Nanocomposites Prepared via Solution Intercalation, Polymer-Plastics Technology and Engineering, 53:10, 1047-1055, DOI: 10.1080/03602559.2014.886068

To link to this article: http://dx.doi.org/10.1080/03602559.2014.886068

PLEASE SCROLL DOWN FOR ARTICLE

Taylor & Francis makes every effort to ensure the accuracy of all the information (the “Content”) contained in the publications on our platform. However, Taylor & Francis, our agents, and our licensors make no representations or warranties whatsoever as to the accuracy, completeness, or suitability for any purpose of the Content. Any opinions and views expressed in this publication are the opinions and views of the authors, and are not the views of or endorsed by Taylor & Francis. The accuracy of the Content should not be relied upon and should be independently verified with primary sources of information. Taylor and Francis shall not be liable for any losses, actions, claims, proceedings, demands, costs, expenses, damages, and other liabilities whatsoever or howsoever caused arising directly or indirectly in connection with, in relation to or arising out of the use of the Content.

This article may be used for research, teaching, and private study purposes. Any substantial or systematic reproduction, redistribution, reselling, loan, sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden. Terms & Conditions of access and use can be found at http://www.tandfonline.com/page/terms-and-conditions
Structural Characterization and Thermal Properties of Chiral Poly(amide-imide)/Modified MgAl Layered Double Hydroxide Nanocomposites Prepared via Solution Intercalation

Shadpour Mallakpour1,2 and Mohammad Dinari1,2

1Organic Polymer Chemistry Research Laboratory, Department of Chemistry, Isfahan University of Technology, Isfahan, Islamic Republic of Iran
2Nanotechnology and Advanced Materials Institute, Isfahan University of Technology, Isfahan, Islamic Republic of Iran

Intercalated poly(amide-imide) (PAI)/modified layered double hydroxide (LDH) nanocomposites (NCs) were synthesized by the solution intercalation method. Modified LDH was synthesized from the co-precipitation reaction of the Al(NO3)3⋅9H2O, Mg(NO3)2⋅6H2O and N,N’-(pyromellitoyl)-bis-valine under ultrasound irradiation. An optically active amino acid containing PAI was synthesized by direct polymerization reaction of chiral diacid and 3,5-diamino-N-(thiazole-2-yl)benzamide under green condition. The synthesized materials were characterized by X-ray diffraction, Fourier transform infrared spectroscopy, thermogravimetric analysis (TGA), field emission scanning electron microscope (FE-SEM) and transmission electron microscopy (TEM) techniques. TEM and FE-SEM analysis confirmed that modified LDH was dispersed within the polymer matrix in intercalated and exfoliated forms.

Keywords Layered double hydroxides; Nanocomposite; Poly(amide-imide); (S)-Valine amino acid; X-ray diffraction

INTRODUCTION

Inorganic/polymer nanocomposites (NCs) are a class of composites in which the inorganic phase dimensions are in the order of nanometers. These NCs contain only a few percent of well-dispersed inorganic components of nanometer size in an organic polymer, and exhibit better properties than pure polymers[1-6]. The most part of previous studied has been focused on NCs based on clay and layered silicates because the starting clay materials are easily available and their intercalation chemistry has been studied for a long time[7-11].

Recently, polymer/layered double hydroxide (LDH) NCs have been studied extensively for their remarkable improvement in the mechanical properties, thermal stability, reduced gas permeability, and flame retardancy compared with virgin polymers and traditional organic–inorganic composites[12-17]. This is mainly due to the effective dispersion of LDH layers of nanometer thickness with the aspect ratios of about 30–100 within the polymer matrix[18].

The LDHs can be represented by the ideal formula [M2+1-x M3+ x(OH)2][(An-)x/n ⋅ mH2O], where M2+ and M3+ represent di- and trivalent metal ions within the brucite-like layers respectively, and An- is an interlayer anion[19-22]. The use of LDH as nanofillers is met with some limitations. The high charge density of LDH layers and the high content of anionic species and water molecules result in strong interlayer electrostatic interactions between the sheets and pronounced hydrophilic properties. So, monomer or polymer cannot easily penetrate within LDH layers nor can LDH layers be easily homogeneously dispersed within hydrophobic polymer matrix.

To ease the intercalation of LDH within the polymer matrix and to achieve a good degree of dispersion, the interlayer space should be modified with suitable, usually organic, anions with intention of increasing both the interlayer distance and the hydrophobicity of LDH layers. The anions are typically intercalated into LDH interlayers by three experimental approaches. The first is the co-precipitation method, which requires the addition of an M2/M3 metal salt solution to a base solution of the desired anions[23-27]. The second is the direct ion exchange method, in which LDHs are stirred in a solution of desired anions of a suitable concentration[27-29]. The third is the rehydration method in which calcined LDH is added to solutions of desired anions[30-32].
Poly(amide-imide)s (PAIs) are a class of high performance polymeric materials with good compromise between thermal stability and processability. They possess desirable characteristic for a variety of applications as they retain good mechanical properties at high temperature by comparable properties with aromatic polyamides and poliamides\(^{33-37}\). PAIs containing L-amino acid linkages can be a good qualification for these purposes because of their important properties such as thermo stable, mechanical strength, good chemical resistance, and high biodegradability. In addition presence of L-amino acids into PAIs backbones causes to have chiral property\(^{38,39}\). Recently molten tetrabutyl ammonium bromide (TBAB) was used as a green solvent for the preparation of PAIs\(^{40}\). Ionic liquids (ILs) are frequently considered as green solvents due to their small vapor pressure and other properties, such as excellent ability to dissolve organic compounds, salts and metals, facile recyclabilities non-flammability and high temperature stability\(^{41-43}\).

In this study, novel NCs of high performance PAI with thiazole side chain and different amount of modified LDH with chiral dicarboxylic acid (2, 4 and 8%) were synthesized for the first time. An optically active amino acid containing diacid was synthesized in high yield from the reaction of pyromellitic dihydride with L-valine amino acid and then it was used for organo-modification of chiral LDH as well as for preparation of thermally stable PAI. Modified-LDH was synthesized by ultrasonic technique in a short time. High performance and optically active PAI was synthesized by the direct polymerization reaction of 3,5-diamino-N-(thiazole-2-yl)benzamide and amino acid containing diacid in green media by using TBAB as molten IL. Different NCs of PAI and modified-LDH were prepared through solution intercalation technique. The structure and morphology of the novel materials was confirmed via different techniques.

### EXPERIMENTAL Materials

All chemicals with analytical reagent were purchased from Fluka Chemical Co. (Buchs, Switzerland), Aldrich Chemical Co. (Milwaukee, WI), Riedel-deHaen AG (Seelze, Germany), and Merck (Darmstadt, Germany). Aluminum nitrate nonahydrate [Al(NO\(_3\))\(_3\)-9H\(_2\)O], magnesium nitrate hexahydrate [Mg(NO\(_3\))\(_2\)-6H\(_2\)O], pyromellitic dihydride (PMDA), sodium hydroxide (NaOH), 2-aminothiazole, 3,5-dinitrobenzoyl chloride, propylene oxide, acetone, hydrazine monohydrate, FeCl\(_3\), L-valine amino acid, glacial acetic acid, TBAB and triphenyl phosphate (TPP) were used as received without further purification. \(N,N'\)-Dimethylformamide (DMF) and \(N,N'\)-dimethylacetamide (DMAc) as solvent were distilled over barium oxide under reduced pressure before use.

### Ultrasonic Assistance Synthesis of MgAl – CO\(_3\)^{2-} and Chiral LDH-Diacid

The reaction was occurred on a MISONIX ultrasonic liquid processor, XL-2000 series with a wave of frequency 2.25 \(\times\) 104 Hz and power 100 W.

Synthesis of LDH – CO\(_3\)^{2-} was carried out via a similar procedure as our earlier report\(^{44}\). For chiral modified LDH; at first, \(N,N'\)-(pyromellitoil)-bis-L-valine diacid was synthesized according to our previous study\(^{45}\). Intercalation of dicarboxylate anions into LDH was carried out as follows; solution A was prepared by dissolving Mg(NO\(_3\))\(_2\)-6H\(_2\)O, Al(NO\(_3\))\(_3\)-9H\(_2\)O in 50 mL of deionized water (Mg/Al molar ratio of 2:1) and solution B was prepared by dissolving NaOH (0.02 mol) and \(N,N'\)-(pyromellitoil)-bis-L-valine (0.02 mol) in 20 mL of deionized water. Then, solution A was dripped into solution B and stirred at R.T for 1 h. Then the pH of resulting suspension was adjusted to pH = 9.0 by adding 1 M NaOH solution. The mixed solution was covered with a stopper and transferred immediately into an ultrasound tool and was sonicated for 1 h. The obtained precipitate was filtered and washed by deionized water and then was dried at 60°C for 24 h.

### Synthesis of Diamine with Thiazole Side Group

3,5-Diamino-N-(thiazole-2-yl)benzamide was synthesized according to our previous study\(^{46}\).

### Synthesis of Optically Active and High Performance PAI

The PAI was prepared by the following general procedure: A mixture of chiral diacid \(N,N'\)-(pyromellitoil)-bis-L-valine (0.20 g, 4.8 \(\times\) 10\(^{-4}\) mol), 3,5-diamino-N-(thiazole-2-yl) benzamide (0.10 g, 4.8 \(\times\) 10\(^{-4}\) mol) and TBAB (0.58 g, 1.8 \(\times\) 10\(^{-3}\) mol) was ground until a powder was formed. After the mixture was completely ground, it was transferred into a 25 mL, round-bottom flask and then 0.28 mL (8.9 \(\times\) 10\(^{-4}\) mol) of TPP was added to the mixture, which was heated until a homogeneous solution was formed. Then, the solution was stirred for 12 h at 120°C, and the viscous solution was precipitated in 10 mL of methanol. The white solid was filtered off and dried to give 0.26 g (89%) of PAI. The specific rotation and inherent viscosity of the synthesized PAI were \(\lbrack\alpha\rbrack_0^D = -26.16^o\) and 0.47 dL/g, respectively (measured at a concentration of 0.5 g dL\(^{-1}\) in DMF at 25°C).

FT-IR (KBr, cm\(^{-1}\)): 3,500 (m, br), 3105 (w), 2910 (w), 1780 (m), 1,723 (s), 1,620 (m), 1,552 (s), 1,455 (s), 1,382 (s), 1,348 (s), 1,317 (s), 980 (s), 965 (m), 804 (m), 756 (m), 720 (m), 680 (m), 550 (m), \(^1\)H-NMR (400 MHz, DMSO-\(d_6\), ppm): 1.03 (d, 12H, CH3, distorted), 2.80 (m, 2H, CH), 4.64-4.65 (d, 2H, CH, \(J = 3.6\) Hz), 6.94 (s, 1H, Ar-H), 7.49 (s, 2H, Ar-H), 7.89-7.91 (d, 1H, Ar-H, \(J = 6.4\) Hz), 8.09-8.11 (d, 1H, Ar-H, \(J = 8.4\)), 8.34 (s, 2H, Ar-H), 10.27 (s, 2H, NH), 12.67 (s, 1H, NH).
Preparation of the PAI/Modified-LDH NCs

NC materials were prepared by mixing the appropriate amounts of the optically active PAI and chiral modified LDH in DMAc as a solvent in a flask for a particular concentration. NCs were prepared by mixing different amounts of modified LDH (2, 4 and 8 wt.%) to the PAI solution. The solution was agitated to high speed stirring at 80°C for 2 h, at 60°C for 2 h and then at 40°C for 20 h for uniform dispersion of modified LDH in the PAI matrix. NC films of uniform thickness were obtained by pouring the hybrid solutions into Petri dishes, followed by solvent evaporation at high temperature. These films were further dried at 80°C under reduced pressure to a constant weight.

Characterization Techniques

Fourier transformed infrared (FT-IR) spectra for pristine LDH and other samples were recorded for wave numbers 4000-400 cm⁻¹ using a Jasco-680 FT-IR spectrometer (Japan). The powdered samples were mixed with KBr and pressed in the form of pellets for the measurement. Band intensities are assigned as weak (w), medium (m), shoulder (sh), strong (s), and broad (br).

Specific rotation was measured with a Jasco (Osaka, Japan) P-1030 polarimeter at the concentration of 0.5 g dL⁻¹ at 25°C. The inherent viscosities of the polymer was determined with a Cannon-Fenske Routine Viscometer (Germany).

X-ray diffraction analysis (XRD) over 2θ = 1.8 to 70°, in steps of 0.02° was carried out using a X-ray diffractometer (Bruker, D8 Avance, Germany) with Cu-Kα radiation (λ = 0.154 nm, monochromatization by primary graphite crystal) generated at 100 mA and 45 kV. XRD curves were interpreted with respect to the position of the basal reflection (003), which depends on the distance between two adjacent metal hydroxide sheets in the LDH lattice. The d-spacing of the sample was analyzed by using Bragg’s equation (nλ = 2d sin θ), where n is an integer, λ is the wavelength, θ is the glancing angle of incidence, and d is the interplanar spacing of the crystal.

Thermogravimetric analysis (TGA) data for samples were taken on a STA503 TA instrument (USA) in a nitrogen atmosphere at a rate of 10°C/min from ambient temperature to 800°C at nitrogen atmosphere.

The morphology of the LDH and NCs was examined by field emission scanning electron microscopy [FE-SEM, HITACHI, S-4160, Japan]]. The powdered sample was dispersed in H₂O, and then the sediment was dried at room temperature before gold coating.

The dispersion of the nanoclay within the medium has been controlled by transmission electron microscopy [TEM, (Philips, CM120, Netherlands)] using an accelerator voltage of 100 kV. For TEM sample preparation, NC was suspended in water and a small drop of it was deposited and dried on a carbon coated copper grid. The inorganic components appear black/grey colored on the micrographs.

RESULTS AND DISCUSSION

Synthesis of Modified-LDH and Preparation of PAI/Modified-LDH NCs

In recent years, polymer/LDH NCs have attracted considerable interest in the field of material chemistry. However, LDH layers possess a high charge density (~300 mequiv/100 g) thus strong interlayer electrostatic interaction makes the exfoliation of LDH much more unfavorable. In addition, pristine LDH is not suitable for the penetration of giant polymer chains or chain segments into their gallery space unless its interlayer distance is significantly increased. Accordingly, the intercalation of LDH involves organic modification of LDH to expand the basal spacing and/or ionogenic modification of the polymer to graft anions onto the polymers or monomers. In this study an optically active diacid was synthesized from the reaction of PMDA and valine amino acid (Scheme 1).

This compound exhibited specific rotations ([α]D25 = -67.2°; measured at a concentration of 0.5 g/dL in DMF at 25°C). It was used for preparation of novel modified-LDH via one pot co-precipitation reaction using ultrasonic wave for the first time. The obtained modified-LDH is organophilic and the interlayer space of this compound is increased (Scheme 1). These phenomena causes enlarge in the d-spacing which facilitates the entry of the host polymer molecules between the layered of modified LDH for further applications of this compound in the synthesis of LDH/polymer NCs.

Molten TBAB was used as solvent for the production of the organo-soluble and thermally stable PAI by the direct polymerization reaction of diacid and diamine with thiazole side chain (Scheme 2). The chiral polymer was synthesized under conventional heating in the presence of TBAB and TPP, and it took 12 h at 120°C for the completion of the reaction and the inherent viscosity of this PAI was 0.47 dL/g. The incorporation of a chiral unit into the polymer backbone was obtained by measuring the specific rotations of [α]D25 = -26.16 (0.05 g in 10 mL of DMF). Chemical structure and purity of this polymer was confirmed in our previous study.[46]

Characterization

FT-IR Study

The LDH-CO₃²⁻ and diacid modified-LDH were confirmed by examination of the FT-IR spectra shown in Figure 1. The spectra of both materials show very broad bands centered at above 3500 cm⁻¹, relating to the ν(OH) stretching vibration of hydroxyl groups of the host layers and to the interlayer and physically absorbed water molecules. For LDH-CO₃²⁻, free CO₃²⁻ anions that
there are in solution show three different FT-IR vibration modes: at 880 cm\(^{-1}\) (bending non-planar mode), 1415 cm\(^{-1}\) (the asymmetric stretching mode) and 680 cm\(^{-1}\) (the bending angular mode). The bending vibration of the interlayer H\(_2\)O is also reflected in the broad bands around 1590 cm\(^{-1}\) and bands due to MgAl–OH translation modes are recorded below 600 cm\(^{-1}\) (Fig. 1)[47].

The FT-IR spectra of modified-LDH show two types of bands: one corresponding to the anionic species intercalated and other corresponding to the host LDH materials (Fig. 1). The absorption bands at 2930–3100 cm\(^{-1}\) correspond to the \(\nu_{asym}\) and \(\nu_{sym}\) (C-H) modes of CH\(_2\) group in the chiral dicarboxylate molecules. The other dicarboxylate bands originated from various functional groups are also found at 1740–1700 cm\(^{-1}\) (C=O), 1300–1000 cm\(^{-1}\) (C-O), and 1600–1475 cm\(^{-1}\) (conjugate C=C). This result indicates the presence of the diacid in the interlayer space of LDH.

Figure 2 shows the FT-IR spectra of the neat PAI and NC of the PAI with 2, 4, and 8% of modified-LDH. The FT-IR spectra of pure PAI showed absorptions around 3360 cm\(^{-1}\) (N–H), 3100 cm\(^{-1}\) (aromatic C–H stretching), 2930 cm\(^{-1}\) and 2857 cm\(^{-1}\) (CH\(_2\) stretching) and two overlapped carbonyl (amide and imide s C=O) absorptions at 1776, 1725, and 1663 cm\(^{-1}\), respectively. Absorption at 1380 and 727 cm\(^{-1}\) showed the presence of the imide heterocycle in this polymer. Compared with pure PAI, a new broad absorption band below 600 cm\(^{-1}\) can be found in the FT-IR spectra of PAI/modified-LDH NCs, which increases with increasing modified-LDH contents and can be ascribed to the vibration of M–O–M groups. From these figures, it can be concluded that the resulting NC materials not only have characteristic neat PAI bands, but also have characteristic peaks for modified-LDH (Fig. 2).

**X-Ray Diffraction**

The powder XRD patterns for the materials prepared are in Figure 3. For LDH-CO\(_3\)^{2−}, a serial of sharp peaks are observed. The gallery spacing between LDH platelets related to the peak of (003) plane (characteristic peak at \(2\theta = 11.84\)) is calculated as around 0.79 nm by Bragg equation. Other peaks at relatively high \(2\theta\) value are indexed to non-basal (006), (009), (110), and (113) reflections[48]. As expected, the position of the basal reflections of modified sample is shifted to higher \(d\) value indicating the expansion in the interlayer distance (Fig. 3). The modified sample does not show a distinct reflection at \(d = 0.76\) nm. The XRD patterns of the modified LDH showed the expanding LDH structure with a sharp (003) spacing of 1.55 nm in

SCH. 1. Synthesis of bioactive diacid and preparation of chiral MLDH.

SCH. 2. Preparation of chiral PAI with thiazole pendant group.
Figure 4. This value indicates that an expansion of the interlayer spacing, compared to the LDH-CO$_3$ ($d_{003} = 0.79$ nm) may be caused by the chiral diacid intercalation into LDH by co-precipitation method.

The XRD patterns of pure PAI and NCs of PAI with modified-LDH (2, 4 and 8%) are shown in Figure 3. In the XRD pattern of the neat PAI, no sharp diffraction peaks was observed and this indicated that this polymer is amorphous. For NCs, the disappearance of the diffraction peaks corresponding to the LDH was observed. The disappearance of LDH peaks in the NCs may be as a result of the partial exfoliated structure, in which the gallery height of intercalated layers is large enough and the layer correlation is not detected by XRD (Fig. 3). Although XRD provides a partial picture about distribution of nanofiller and disappearance of the peak corresponding to $d$-spacings does not always confirm the exfoliated NCs, a complete characterization of NC morphology requires microscopic investigation.

**FE-SEM**

The FE-SEM micrograph of LDH-CO$_3$ and modified-LDH are shown in Figure 4. The FE-SEM image of pure LDH reveals the nature of LDH particles, which roughly consists of plate-like shape stacked on top of each other (Figs. 4a and 4b). The FE-SEM images of the modified-LDH are similar to the unmodified LDH and the particle sizes of the nanolayers are between 20–50 nm (Figs. 4c and 4d).

The surface morphology of the PAI/modified-LDH NCs was also investigated by FE-SEM technique as shown
in Figure 5. According to these images, in NC4%, the LDH nanosheets have a better dispersion in comparison with NC with high amount of modified-LDH (8%).

**TEM**

The most direct measure of the dispersion of these Mg/Al nanolayers in PAI matrix is typically TEM micrographs of the PAI/modified LDH NCs. Typical TEM images of modified-LDH and NC of PAI and 4 wt% of modified-LDH are shown in Figures 6a–6d. In the TEM micrograph of the modified-LDH, the platelets have a hexagonal shape with rounded corners. There are no signs of aggregation visible in the micrographs (Figs. 6a and 6b). TEM images of the NC4% show that the modified-LDHs were dispersed in the polymer matrix by intercalated and exfoliated states (Figs. 6c and 6d). These results of TEM are consistent with the XRD.

**Thermal Degradation Characteristics**

The thermal behavior of the LDH-CO3 and modified-LDH was examined by TGA technique and the curves are shown in Figure 7. For the LDH-CO3 the TGA curve shows three weight loss steps which relate to the desorption of absorbed water at around 104°C, the dehydroxylation of the brucite-like layer at around 230°C as well as the decomposition of carbonate ions at around 350–400°C (Fig. 7). The organic modification of the LDH changes its the thermal decomposition behavior in comparison to the unmodified sample[29,44].

As can be seen in Figure 7, the numbers of decomposition stages have increased for modified-LDH. Also degradation of interlayer diacid anion took place before de-hydroxylation process in the case of modified LDHs and so showed less thermal stability than the original LDH. The presence of a larger weight loss step within 370–560°C for LDH-dicarboxylate anions compared to the LDH confirmed the presence of interlayer surfactant anions in LDH.

The thermo-stability of the pure amino acid containing PAI and the NCs of PAI and different amount of modified-LDH (2, 4, and 8%) were evaluated by TGA under nitrogen atmosphere at a heating rate of 10°C min−1, and

![FIG. 6. TEM micrographs of modified LDH (a and b) and NC4% (c and d).](image)

![FIG. 7. TGA thermograms of LDH and modified LDH with chiral dicarboxylate.](image)
the corresponding weight loss temperatures of 5% and 10% ($T_{5\%}$ and $T_{10\%}$) were all determined from original TGA curves. Typical TGA curves of representative samples are shown in Figure 8. In addition, the results obtained from these analyses are completely tabulated in Table 1. Single-step degradation was observed in all samples.

The onset of decomposition temperature of the NCs was higher than that of pure PAI, shifting toward higher temperatures as the amount of modified-LDH was increased. The marginal increase in thermal stability of PAI matrix upon incorporation of modified-LDH could be attributed to the higher thermal conductivity of modified-LDH that facilitated heat dissipation within the composites, hence preventing the accumulation of heat at certain points for degradation. The end temperature of decomposition was also retarded with increasing modified-LDH content.

The masses remaining at 800°C were almost entirely due to the remaining modified-LDH, consistent with initial modified-LDH loading. The weight percent remaining after major degradation at 800°C was higher for composites rather than neat PAI. This indicated that modified-LDH reduced the degradation of PAI at high temperature as the effect was clearly seen in the curves. Therefore, it could be demonstrated that a small amount of modified-LDH acted as effective thermal degradation resistant reinforcement in the PAI matrix, increasing the thermal stability of the PAI/modified-LDH NCs [29].

CONCLUSIONS

The very exciting recent progress in intercalation reactions of LDHs shows that this area of existing solid state chemistry has a very promising future. Especially, the continuing work on the intercalation of biologically active guests has great potential. In the work, a new organic-inorganic hybrid material has been prepared in a simple, low cost, efficient, and environmentally-friendly method by one-pot co-precipitation reaction of the Al(NO$_3$)$_3$·9H$_2$O, Mg(NO$_3$)$_2$·6H$_2$O and bioactive N,N’-(pyromellitoyl)-bis-L-valine under ultrasonic irradiation in aqueous media.

The modified Mg-Al LDH shows an increase in interlayer distance as compared to the unmodified LDH by XRD. Chiral amino acid containing high performance PAI with thiazole side chain was prepared in molten TBAB as a green solvent. Then different NCs of PAI and modified-LDH were prepared by a solution-intercalation method. XRD and FT-IR results pointed to the incorporation of the modified-LDH within the PAI matrix. FE-SEM and TEM analysis demonstrate that the exfoliated and partially exfoliated NCs are formed. Thermal stability of the PAI/modified-LDH NCs is increased by about 4–10°C compared with neat PAI.

FUNDING

We wish to express our gratitude to the Research Affairs Division Isfahan University of Technology (IUT), Isfahan, for partial financial support. Further financial support from National Elite Foundation (NEF), Iran Nanotechnology Initiative Council (INIC) and Center of Excellency in Sensors and Green Chemistry Research (IUT) is gratefully acknowledged.

REFERENCES

4. Naffakh, M.; Diez-Pascual, A.M.; Marco, C.; Ellis, G.J.; Gómez-Fatou, MA. Opportunities and challenges in the use of inorganic

![FIG. 8. TGA thermograms of PAI and different PAI/modified-LDH NCs.](image-url)


29. Mallakpour, S.; Dinari, M. Facile synthesis of nanocomposite materials by intercalating an optically active poly(amide-imide) enclosing (L)-isoleucine moieties and azobenzene side groups into a chiral layered double hydroxide. Polymer 2013, 54, 2907–2916.


43. Smith, T.W.; Zhao, M.; Yang, F.; Smith, D.; Cebe, P. Imidazole polymers derived from iodine liquid 4-vinylimidazolium monomers:
Their synthesis and thermal and dielectric properties. Macromolecules 2013, 46, 1133–1143.


