

## Documents

Azahari, N.N.A.<sup>a</sup>, Anuar, H.<sup>a</sup>, Hassan, A.<sup>b</sup>, Jawaid, M.<sup>c</sup>, Halim, Z.<sup>a</sup>, Samsudin, S.A.<sup>d</sup>

### **Thermal, Dynamic Mechanical, Mechanical and Flammability Properties of Halloysite Nanotubes Filled Polyamide 11 Nanocomposites**

(2023) *Malaysian Journal of Fundamental and Applied Sciences*, 19 (2), pp. 173-193.

**DOI:** 10.11113/mjfas.v19n2.2684

<sup>a</sup> Department of Manufacturing and Materials Engineering, Kulliyyah of Engineering, International Islamic University Malaysia, Gombak, Kuala Lumpur, 53100, Malaysia

<sup>b</sup> School of Bioprocess and Polymer Engineering, Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia, Johor, Johor Bahru, 81310 UTM, Malaysia

<sup>c</sup> Department of Biocomposite Technology Lab, Institute of Tropical Forestry and Forest Product, Universiti Putra Malaysia, Selangor, Serdang, 43400, Malaysia

<sup>d</sup> School of Chemical Engineering and Energy Engineering, Faculty of Engineering, Johor Bahru, Johor, 81310 UTM, Malaysia

#### **Abstract**

The effects of various filler contents on the thermal, dynamic mechanical, mechanical, as well as flammability properties of halloysite nanotubes (HNTs) filler and polyamide 11 (PA 11) matrixes are investigated in this research. The nanocomposites were made out of 100 phr of PA 11 and three distinct HNTs loadings of 2, 4, and 6 phr each. PA 11 nanocomposites without HNTs filler was used as the reference sample. To melt-compound the nanocomposites, a twin-screw extruder was used, and the specimen for testing was then injected using an injection mold. SEM, TGA, DSC, FTIR, DMA, tensile, flexural, impact, and UL-94 flammability tests were conducted on the nanocomposites. Incorporation of 4 phr HNTs into the nanocomposites resulted in the highest tensile and flexural strength. Maximum improvement in the DMA, Young's and flexural modulus was achieved at 6 phr HNTs content. The elongation at break and TGA resulted the highest increase at 2 phr HNTs content. However, the impact strength decreased with increasing HNTs content. Scanning electron microscopy revealed the ductility of the nanocomposites with increased HNTs content up to 4 phr. The DSC showed a steady increase in melting temperature ( $T_m$ ) as HNTs content increased up to 4 phr, while the crystallization temperature ( $T_c$ ) remained unchanged. TGA of PA 11/HNTs nanocomposites showed high thermal stability at 2 phr HNTs content. However, on further addition of HNTs up to 6 phr, thermal stability of the nanocomposites decreased due to the excess amount of HNTs. All the nanocomposites passed the horizontal and vertical UL-94 test with HB and V-2 grade. PA 11/4HNTs nanocomposite has the highest tensile strength, flexural strength compared to other PA 11/HNTs nanocomposites. PA 11/4HNTs nanocomposite can be suggested as an optimum formulation with balanced mechanical properties in terms of toughness. © 2023 The Korean Association for Radiation Protection.

#### **Author Keywords**

flammability properties; Halloysite nanotubes; mechanical properties; nanocomposites; Polyamide

#### **References**

- Bugatti, V., Vertuccio, L., Viscusi, G., Gorrasi, G.  
**Antimicrobial membranes of bio-based pa 11 and hnts filled with lysozyme obtained by an electrospinning process**  
(2018) *Nanomaterials*, 8 (3).
- Androsch, R., Jariyavidyanont, K., Schick, C.  
**Enthalpy relaxation of polyamide 11 of different morphology far below the glass transition temperature**  
(2019) *Entropy*, 21 (10), p. 984.
- Fu, S., Sun, Z., Huang, P., Li, Y., Hu, N.  
**Some basic aspects of polymer nanocomposites: A critical review**  
(2019) *Nano Mater. Sci.*, 1 (1), pp. 2-30.
- Bidsorkhi, H. C., Adelnia, H., Heidar Pour, R., Soheilmoghaddam, M.  
**Preparation and characterization of ethylene-vinyl acetate/halloysite nanotube nanocomposites**  
(2015) *J. Mater. Sci.*, 50 (8), pp. 3237-3245.

- Mousa, M., Dong, Y.  
**(2021) Multiscaled PVA Bionanocomposite Films,**
- Ng, C. L., Chow, W. S.  
**Multifunctional halloysite nanotube-reinforced polypropylene/polyamide binary nanocomposites**  
(2020) *Polym. Polym. Compos.*, 28 (8-9), pp. 623-630.
- Arman, N., Tekay, E., Şen, S.  
**Preparation of high-strength SEBS nanocomposites reinforced with halloysite nanotube: Effect of SEBS-g-MA compatibilizer**  
(2020) *J. Thermoplast. Compos. Mater.*, 33 (10), pp. 1336-1357.
- Marset, D.  
**The effect of halloysite nanotubes on the fire retardancy properties of partially biobased polyamide 610**  
(2020) *Polymers (Basel)*, 12 (12), pp. 1-21.
- Kovačević, Z., Flinčec Grgac, S., Bischof, S.  
**Progress in biodegradable flame retardant nano-biocomposites**  
(2021) *Polymers (Basel)*, 13 (5), pp. 1-30.
- Prashantha, K., Lacrampe, M. F., Krawczak, P.  
**Highly dispersed polyamide-11/halloysite nanocomposites: Thermal, rheological, optical, dielectric, and mechanical properties**  
(2013) *J. Appl. Polym. Sci.*, 130 (1), pp. 313-321.
- Francisco, D., de Paiva, L., Aldeia, W., Lugao, A., Moura, E.  
(2019) *Investigation on mechanical behaviors of polyamide 11 reinforced with halloysite nanotubes*, pp. 693-701.  
Springer International Publishing
- Sahnoune, M.  
**Tribological and mechanical properties of polyamide-11/halloysite nanotube nanocomposites**  
(2019) *J. Polym. Eng.*, 39 (1), pp. 25-34.
- Sahnoune, M., Kaci, M., Garay, H., Lopez-Cuesta, J. M., Mahlous, M.  
**Effects of gamma irradiation on structural, thermal and mechanical properties of polyamide-11/halloysite nanotubes nanocomposites**  
(2021) *J. Thermoplast. Compos. Mater.*,
- Dong, Y., Bhattacharyya, D.  
**Effects of clay type, clay/compatibiliser content and matrix viscosity on the mechanical properties of polypropylene/organoclay nanocomposites**  
(2008) *Compos. Part A Appl. Sci. Manuf.*, 39 (7), pp. 1177-1191.
- Fukushima, K., Tabuani, D., Camino, G.  
**Nanocomposites of PLA and PCL based on montmorillonite and sepiolite**  
(2009) *Materials Science and Engineering C*, 29 (4), pp. 1433-1441.
- Girard, E., Liu, X., Marty, J. D., Destarac, M.  
**RAFT/MADIX (co)polymerization of vinyl trifluoroacetate: A means to many ends**  
(2014) *Polym. Chem.*, 5 (3), pp. 1013-1022.
- Kolesov, I.  
**Crystallization of a polyamide 11/organo-modified montmorillonite nanocomposite at rapid cooling**  
(2013) *Colloid Polym. Sci.*, 291 (11), pp. 2541-2549.

- Sahnoune, M.  
*(2020) Effects of functionalized halloysite on morphology and properties of polyamide-11 / SEBS-g-MA blends To cite this version,*  
HAL Id: hal-02892647
- Thompson, N. B. A.  
**The thermal decomposition of studtite: analysis of the amorphous phase**  
(2021) *J. Radioanal. Nucl. Chem.*, 327 (3), pp. 1335-1347.
- Zhang, S., Ran, Q., Zhang, X., Gu, Y.  
**Effects of the curing atmosphere on the structures and properties of polybenzoxazine films**  
(2021) *J. Mater. Sci.*, 56 (3), pp. 2748-2762.
- Loulergue, P., Amela-Cortes, M., Cordier, S., Molard, Y., Lemiègre, L., Audic, J. L.  
**Polyurethanes prepared from cyclocarbonated broccoli seed oil (PUcc): New biobased organic matrices for incorporation of phosphorescent metal nanocluster**  
(2017) *J. Appl. Polym. Sci.*, 134 (45), pp. 1-10.
- Jumahat, A., Talib, A. A. A., Abdullah, A.  
(2016) *Wear Properties of Nanoclay Filled Epoxy Polymers and Fiber Reinforced Hybrid Composites*,
- Crossley, R., Schubel, P., Stevenson, A.  
**Furan matrix and flax fibre as a sustainable renewable composite: Mechanical and fire-resistant properties in comparison to phenol, epoxy and polyester**  
(2014) *J. Reinf. Plast. Compos.*, 33 (1), pp. 58-68.
- Wu, J. H., Chen, C. W., Kuo, M. C., Yen, M. S., Lee, K. Y.  
**High toughness and fast crystallization poly(lactic acid)/polyamide 11/SiO<sub>2</sub> composites**  
(2018) *J. Polym. Environ.*, 26 (2), pp. 626-635.
- Sharif, N. F. A., Mohamad, Z., Hassan, A., Wahit, M. U.  
**Novel epoxidized natural rubber toughened polyamide 6/halloysite nanotubes nanocomposites**  
(2012) *J. Polym. Res.*, 19 (1).
- Arshad, J., Janjua, N. K., Raza, R.  
**Synthesis of novel (Be,Mg,Ca,Sr,Zn,Ni)3O4 high entropy oxide with characterization of structural and functional properties and electrochemical applications**  
(2021) *J. Electrochem. Sci. Technol.*, 12 (1), pp. 112-125.
- Jamaludin, N. A., Inuwa, I. M., Hassan, A., Othman, N., Jawaid, M.  
**Mechanical and thermal properties of SEBS-g-MA compatibilized halloysite nanotubes reinforced polyethylene terephthalate/polycarbonate/nanocomposites**  
(2015) *J. Appl. Polym. Sci.*, 132 (39), pp. 1-10.
- Mrówka, M., Woźniak, A., Nowak, J., Wróbel, G., Ślawski, S.  
**Determination of mechanical and tribological properties of silicone-based composites filled with manganese waste**  
(2021) *Materials (Basel)*, 14 (16).
- Mancic, L., Osman, R. F. M., Costa, A. M. L. M., d'Almeida, J. R. M., Marinkovic, B. A., Rizzo, F. C.  
**Thermal and mechanical properties of polyamide 11 based composites reinforced with surface modified titanate nanotubes**  
(2015) *Mater. Des.*, 83, pp. 459-467.
- Carponcin, D.  
**Discontinuity of physical properties of carbon nanotube/polymer composites at the**

**percolation threshold**

(2014) *J. Non. Cryst. Solids*, 392-393, pp. 19-25.

- Zubkiewicz, A., Szymczyk, A., Paszkiewicz, S., Jędrzejewski, R., Piesowicz, E., Siemiński, J.

**Ethylene vinyl acetate copolymer/halloysite nanotubes nanocomposites with enhanced mechanical and thermal properties**

(2020) *J. Appl. Polym. Sci*, 137 (38), pp. 1-12.

- Jamaludin, N. A., Hassan, A., Othman, N., Jawaid, M.

**Effects of halloysite nanotubes on mechanical and thermal stability of poly(ethylene terephthalate)/polycarbonate nanocomposites**

(2015) *Appl. Mech. Mater*, 735, pp. 8-12.

- Inuwa, I. M., Keat, T. B., Hassan, A.

**Mechanical and thermal properties of hybrid Graphene/Halloysite nanotubes reinforced polyethylene terephthalate nanocomposites**

(2016) *Nanoclay Reinforced Polym. Compos*, pp. 309-327.

Springer, Singapore

- Rojas-Lema, S., Quiles-Carrillo, L., Garcia-Garcia, D., Melendez-Rodriguez, B., Balart, R., Torres-Giner, S.

**Tailoring the properties of thermo-compressed polylactide films for food packaging applications by individual and combined additions of lactic acid oligomer and halloysite nanotubes**

(2020) *Molecules*, 25 (8), pp. 1-23.

- Cheng, Z.-L., Chang, X.-Y., Liu, Z., Qin, D.-Z.

**Surface-modified halloysite nanotubes as fillers applied in reinforcing the performance of polytetrafluoroethylene**

(2018) *Clay Miner*, 53 (4), pp. 643-656.

- Ghanbari, M., Emadzadeh, D., Lau, W. J., Riazi, H., Almasi, D., Ismail, A. F.

**Minimizing structural parameter of thin film composite forward osmosis membranes using polysulfone/halloysite nanotubes as membrane substrates**

(2016) *Desalination*, 377, pp. 152-162.

- Liu, T. X., Chen, D., Phang, I. Y., Wei, C.

**Studies on crystal transition of polyamide 11 nanocomposites by variable-temperature x-ray diffraction**

(2014) *Chinese J. Polym. Sci. (English Ed.)*, 32 (1), pp. 115-122.

- Krishnamoorti, R., Vaia, R. A., Giannelis, E. P.

**Structure and dynamics of polymer-layered silicate nanocomposites**

(1996) *Chem. Mater*, 8, pp. 1728-1734.

- Rawtani, D., Agrawal, Y. K.

**Multifarious applications of halloysite nanotubes: A review**

(2012) *Rev. Adv. Mater. Sci*, 30 (3), pp. 282-295.

- Liu, M., Guo, B., Du, M., Cai, X., Jia, D.

**Properties of halloysite nanotube-epoxy resin hybrids and the interfacial reactions in the systems**

(2007) *Nanotechnology*, 18 (45).

- Du, M., Jia, D.

(2009) *Newly emerging applications of halloysite nanotubes: a review*, pp. 574-582.

June

- Mohamed, N. K., Kochkodan, V., Zekri, A., Ahzi, S.

**Polysulfone membranes embedded with halloysites nanotubes: Preparation and**

**properties**(2020) *Membranes (Basel)*, 10 (1).

- Rashmi, B. J., Prashantha, K., Lacrampe, M. F., Krawczak, P.  
**Toughening of poly(Lactic acid) without sacrificing stiffness and strength by melt-blending with polyamide 11 and selective localization of halloysite nanotubes**  
(2015) *Express Polym. Lett.*, 9 (8), pp. 721-735.
- Prashantha, K., Schmitt, H., Lacrampe, M. F., Krawczak, P.  
**Mechanical behaviour and essential work of fracture of halloysite nanotubes filled polyamide 6 nanocomposites**  
(2011) *Compos. Sci. Technol.*, 71 (16), pp. 1859-1866.
- Thanakkasarane, S., Sadeghi, K., Seo, J.  
**Smart steam release of newly developed temperature-responsive nanocomposite films derived from phase change material**  
(2020) *Polymer (Guildf)*, 219, p. 123543.  
(November)
- Hao, A.  
(2014) *Mechanical, thermal, and flame-retardant performance of polyamide 11 – halloysite nanotube nanocomposites*, pp. 157-167.
- Szpilska, K., Czaja, K., Kudła, S.  
**Halloysite nanotubes as polyolefin fillers**  
(2015) *Polimery/Polymers*, 60 (6), pp. 359-371.
- Khare, H. S., Burris, D. L.  
**A quantitative method for measuring nanocomposite dispersion**  
(2010) *Polymer (Guildf)*, 51 (3), pp. 719-729.
- Deng, S., Zhang, J., Ye, L.  
**Halloysite-epoxy nanocomposites with improved particle dispersion through ball mill homogenisation and chemical treatments**  
(2009) *Compos. Sci. Technol.*, 69 (14), pp. 2497-2505.
- Mishra, G., Mukhopadhyay, M.  
**Enhanced antifouling performance of halloysite nanotubes (HNTs) blended poly(vinyl chloride) (PVC/HNTs) ultrafiltration membranes: For water treatment**  
(2018) *J. Ind. Eng. Chem.*, 63, pp. 366-379.
- Sangeetha, V., Gopinath, D., Prithivirajan, R., Girish Chandran, V., Manoj Kumar, R.  
**Investigating the mechanical, thermal and melt flow index properties of HNTs – LLDPE nano composites for the applications of rotational moulding**  
(2020) *Polym. Test.*, 89, p. 106595.
- Prashantha, K., Lacrampe, M. F., Krawczak, P.  
**Processing and characterization of halloysite nanotubes filled polypropylene nanocomposites based on a masterbatch route: Effect of halloysites treatment on structural and mechanical properties**  
(2011) *Express Polym. Lett.*, 5 (4), pp. 295-307.
- Montava-Jorda, S., Chacon, V., Lascano, D., Sanchez-Nacher, L., Montanes, N.  
**Manufacturing and characterization of functionalized aliphatic polyester from poly(lactic acid) with halloysite nanotubes**  
(2019) *Polymers (Basel)*, 11 (8).
- Franciszczak, P., Taraghi, I., Paszkiewicz, S., Meljon, A., Piesowicz, E., Burzyński, M.  
**Effect of halloysite nanotube on mechanical properties, thermal stability and morphology of polypropylene and polypropylene/short kenaf fibers hybrid**

**biocomposites**(2020) *Materials (Basel)*, 13 (19), pp. 1-13.

- Wu, H., Ortiz, R., Koo, J. H.

**Rubber toughened flame retardant (FR) polyamide 11 nanocomposites Part 1: the effect of SEBS-g-MA elastomer and nanoclay**

(2018) *Flame Retard. Therm. Stab. Mater.*, 1 (1), pp. 25-38.

- Butler, S., Kim, G., Koo, J. H.

**Polyamide 11-Halloysite nanotube nanocomposites: Mechanical, thermal and flammability characterization**

(2011) *Proceedings of the 2011*,

SAMPE ISTC

**Correspondence Address**

Azahari N.N.A.; Department of Manufacturing and Materials Engineering, Gombak, Malaysia; email:

najmatyraazahari@gmail.com

**Publisher:** Penerbit UTM Press**ISSN:** 2289599X**Language of Original Document:** English**Abbreviated Source Title:** Malaysian J. Fundam. App. Sci.

2-s2.0-85158837611

**Document Type:** Article**Publication Stage:** Final**Source:** Scopus ELSEVIER

Copyright © 2023 Elsevier B.V. All rights reserved. Scopus® is a registered trademark of Elsevier B.V.

 RELX Group™