

Optimizing Flexural Strength in Polyester/Luffa Fiber Composites via Gamma Radiation Treatment

Optimización de la fuerza en flexión en compuestos de resina poliéster /fibras de luffa, mediante tratamiento de radiación gamma

H. O. Camacho-Gutiérrez ^{a,*}, G. Martínez-Barrera ^b, L. E. Lugo-Uribe ^c

^{a, b} Laboratorio de Investigación y Desarrollo de Materiales Avanzados (LIDMA), Facultad de Química, Universidad Autónoma del Estado de México, Km. 12 de la carretera Toluca-Atlaquilco, San Cayetano 50200, México.

^c Centro de Tecnología Avanzada (CIATEQ), 52004 Lerma, Estado de México, México

Abstract

This investigation aims to assess the flexural properties of polyester resin/luffa fibers composites. Three different kinds of specimens were tested; the first one consisted of polyester resin exposed to gamma rays at doses ranging from 0 to 500 kGy. The second were composites produced with polyester resin and 1-5 wt% luffa fibers, and the last one consisted of polyester resin and 5% luffa fibers composites exposed to gamma rays (100, 200, 400 and 500 kGy doses). The results show a maximum increase of 27% in flexural modulus when polyester resin was irradiated at a 500 kGy dose. However, there was a decrease of 72% in deformation when 5% of luffa fibers were added. This indicates an increase in rigidity in all three types of specimens but an adverse effect on the strain at rupture. Therefore, the combination of adding luffa fibers and exposing the composites to gamma rays appears to be a suitable method for improving rigidity, albeit with some limitations in deformation.

Keywords: Polyester resin, Luffa fibers, Composites, Gamma radiation, Flexural properties.

Resumen

Esta investigación tiene como objetivo evaluar las propiedades de flexión de los compuestos de resina de poliéster y fibras de luffa. Se probaron tres tipos diferentes de especímenes: el primero consistió en resina de poliéster expuesta a rayos gamma en dosis que variaron de 0 a 500 kGy. El segundo tipo fueron compuestos producidos con resina de poliéster y fibras de luffa en porcentajes de 1-5 en peso, y el último tipo consistió en compuestos de resina de poliéster y fibras de luffa al 5%, expuestos a dosis de rayos gamma de 100, 200, 400 y 500 kGy. Los resultados muestran un aumento máximo del 27% en el módulo de flexión cuando la resina de poliéster fue irradiada a una dosis de 500 kGy. Sin embargo, hubo una disminución del 72% en la deformación cuando se agregó un 5% de fibras de luffa. Esto indica un aumento en la rigidez en los tres tipos de especímenes, pero un efecto adverso en la deformación antes de la ruptura. Por lo tanto, la combinación de agregar fibras de luffa y exponer los compuestos a rayos gamma parece ser un método adecuado para mejorar la rigidez, aunque con algunas limitaciones en la deformación.

Palabras clave: Resina Poliester, fibras de luffa, materiales compuestos, radiación gamma, propiedades de flexión.

1. Introducción

In recent years, using natural fibers as reinforcement in polymer composites has attracted great interest in materials science and engineering. Fibers derived from renewable sources possess remarkable characteristics, such as low cost, low density, and biodegradability, which make them

candidates for improving composite materials' mechanical features and sustainability. Among the natural fibers explored, Luffa cylindrica, commonly known as sponge gourd or luffa, has emerged as a compelling option due to its properties, including high aspect ratio, good tensile strength, and abundant availability (Bal, 2004; Shen, 2012; Joshi, 2004)

*Autor para la correspondencia: gonzomartinez02@yahoo.com.mx

Correo electrónico: hocg6toe@hotmail.com (Héctor O. Camacho-Gutiérrez), gonzomartinez02@yahoo.com.mx (Gonzalo Martínez-Barrera), luis.lugo@ciateq.mx (Luis E. Lugo-Uribe).

Polyester-based composites incorporating Luffa fibers have shown great potential in various applications, such as automotive components, building materials, and consumer goods (Oboh, 2009; Siqueira, 2010). However, achieving optimal mechanical properties remains a key challenge, particularly in terms of flexural strength. A significant problem contributing to this challenge is the aging of polyester resins, which are prone to degrade over time, mainly due to the loss of styrene monomer (Stamatakis, 2010). This aging process can lead to a decrease in the mechanical properties and, consequently, in the long-term durability of composite materials. (Haghdan, 2015; Qazvini, 2005)

To unlock the full potential of polyester resin/natural fiber composites and broaden their applicability, it is imperative to develop innovative techniques that improve their mechanical features while maintaining their eco-friendly and sustainable character. Several processes have been explored to improve the properties of natural fiber-reinforced composites, including chemical and physical treatments. Chemical treatments, such as alkali and silane coupling, have improved adherence between natural fibers and polymer matrices (Hasan, 2022; Saha, 2016). However, sometimes, these treatments use chemicals, which can cause environmental problems. On the other hand, physical treatments include processes such as mechanical fibrillation and heat treatment. They offer advantages in terms of environmental friendliness, simplicity, and mechanical properties, particularly in improvements in flexural strength (Alcañiz, 2022).

Gamma radiation treatment presents distinct advantages compared to other alternatives. It offers controlled and uniform modification without the need of chemical additives. Moreover, it has the potential to enhance fiber-matrix adherence and mitigate the aging effects on the polyester resins, which is a critical concern for composites operating long-term. (Ayma, 2017; Güven, 2016; Zhou, 2015)

When gamma radiation interacts with polyester resins, it initiates a series of chemical reactions within this, sometimes producing cross-linking of polymer chains and increasing its resistance. In addition, gamma rays can induce modifications in microstructure. These features of the gamma radiation process underline its potential for improving composite materials. (Abdel, 2021; Hossain, 2023).

This work studies the flexural strength of polyester resin/luffa fiber composites after exposure to gamma radiation, which can be a solution for the aging problem of polyester resin caused by the gradual loss of styrene monomer. In addition, a viable solution is presented to improve the mechanical characteristics of composite materials, promoting their environmental sustainability in terms of the benefits offered by gamma radiation treatment.

2. Materials and methods

2.1. Materials

A general-purpose orthophthalic polyester resin produced more than two years ago (POLYLITE 33915-15 from the manufacturer REICHHOLD) was used. Luffa cylindrica, often referred to as fluffy gourd, has a robust outer skin and an inner structure with fibers arranged in multiple directions (Figure 1). Fibers no longer than 9 mm in length were extracted from the inner core, cut manually with scissors to avoid possible

deformation caused by friction when subjected to an electrical cutting machine.



Figure 1: Dried Luffa cylindrica fruit.

To ensure fibers length ranging from 5 to 9 mm, an optical microscope was used (Figure 2).

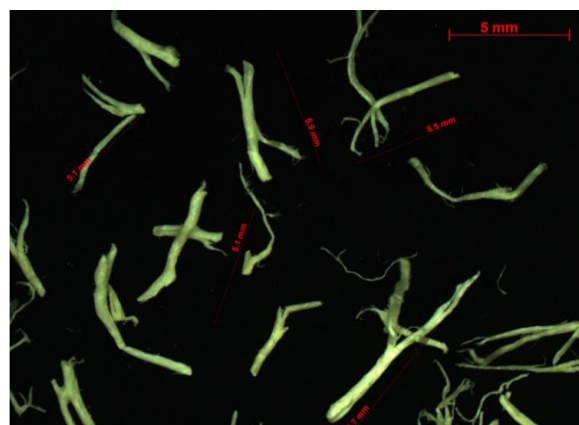


Figure 2: Optical images of Luffa fibers.

2.2. Types of specimens

Three types of specimens were produced, namely:

- Polyester resin irradiated at 100-500 kGy doses.
- Polyester resin/Luffa fibers composites; varying the Luffa fiber concentration from 1% to 5%.
- Polyester resin with 5% luffa fibers, irradiated at 100-500 kGy doses.

For each type of composite, eight specimens were produced.

2.3. Preparation of resin

Due to the age of the resin, the gel time was longer than 2 hours. Therefore, it was necessary to use 0.8% cobalt octoate as a promoter and 1% MEKP as the initiator to reduce the gel time to half an hour.

2.4 Composite production

The composite mixtures were poured into the steel mold following ISO 527 (Figure 3). To avoid the formation of bubbles, the samples were placed in a vibrating bed for 3 minutes.



Figure 3: Molds used for the production of composites.

2.5 Gamma irradiation

The composites were exposed to 100, 200, 300, 400, and 500 kGy doses in a JS-6500 irradiator, which uses cobalt-60 sources, located at the National Institute for Nuclear Research (ININ).

3. Experimental testing

3.1. Flexural testing

The flexural tests were performed in a universal testing machine CONTROLS, equipped with a 30 kN load cell according to ISO 178 (Figure 4). The test parameters are listed in Table 1.

Table 1: Parameters of the flexural tests.

Parameter	Value
Support span, mm	64
Support radius, mm	5
Test speed, mm/min	2
Pre-load, N	0.3
Pre-load speed, mm/min	2



Figure 4: Flexural testing of composite materials.

4. Results and discussion

4.1 Polyester resin

Polyester resin showed a flexural modulus of 2.83 GPa, gradually increasing with the radiation dose (Figure 5 and Table 2). This can be attributed to the cross-linking of polymer chains within polyester resin. The maximal improvement (27%) was obtained for resin irradiated at 500 kGy with respect to non-irradiated resin.

The maximum flexural strength of the resin was 97.0 MPa, which was not significantly modified after irradiation. This improved by 6% for an irradiation dose of 300 kGy (Figure 5b).

The deformation varies gradually after increasing the irradiation dose. The non-irradiated resin shows a value of 4.9%, decreasing up to 2.5% after exposure to 500 kGy. This means 48% less. Gamma rays produced cross-linking of polymer chains, generating a stiffness increase in the resin and, consequently, lower deformation (Figure 5c).

Table 2: Results of polyester resin.

Dose (kGy)	Flexural modulus (GPa)	Maximum flexural strength (MPa)	Strain at rupture (%)
0	2.83	97.0	4.9
100	3.08	96.4	3.2
200	3.33	100.5	3.0
300	3.35	102.9	3.0
400	3.57	102.2	2.9
500	3.61	95.5	2.5

4.2 Polyester resin and luffa fibers

The increase of the Luffa concentration modifies the flexural modulus, having two well-defined stages. The first one for composites with 1 to 3% luffa fibers, at this point, reaches 3.56 GPa compared to that for pure resin. This means 25% more. However, for higher fiber contents, the values go down (Figure 6a and Table 3).

The addition of Luffa fibers drastically affects the maximum flexural strength since the value for pure resin (97 MPa) decreases up to 35.7 MPa, 63% less. Thus, stress transfer between the polymer matrix and fibers is modified, becoming a more fragile resin (Figure 6b).

Similar behavior was obtained for the strain at rupture values since the value for resin (4.4%) was severely affected when adding more fiber concentrations, getting a decrease of 72% with the pure resin value (Figure 6c).

Table 3: Results of polyester resin/Luffa fibers composites.

Luffa (%)	Flexural modulus (GPa)	Maximum flexural strength (MPa)	Strain at rupture (%)
0	2.83	97.0	4.4
1	2.97	47.3	1.7
2	3.11	46.8	1.5
3	3.56	47.5	1.4
4	3.11	47.3	1.3
5	3.09	35.7	1.2

4.3 Polyester resin with 5% of luffa fibers exposed to radiation.

These composites show an increase almost constant in the flexural modulus for expositions at 100-400 kGy, varying by 5% among them. Nevertheless, a notable increase is obtained at 500 kGy dose since the modulus was 3.54 GPa at this dose,

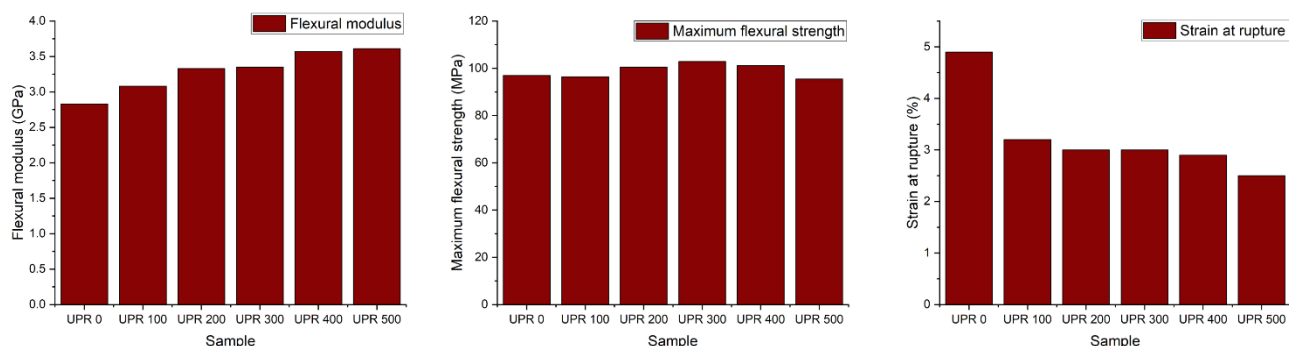


Figure 5: Non-irradiated and irradiated polyester resin: (a) Flexural modulus, (b) Maximum flexural strength, (c) Strain at rupture

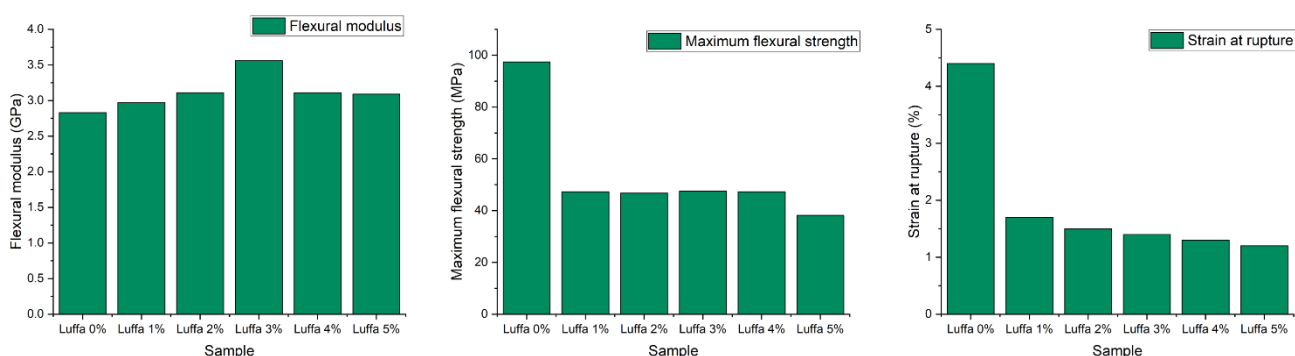


Figure 6: Polyester resin/luffa fibers composites: (a) Flexural modulus, (b) Maximum flexural strength, (c) Strain at rupture

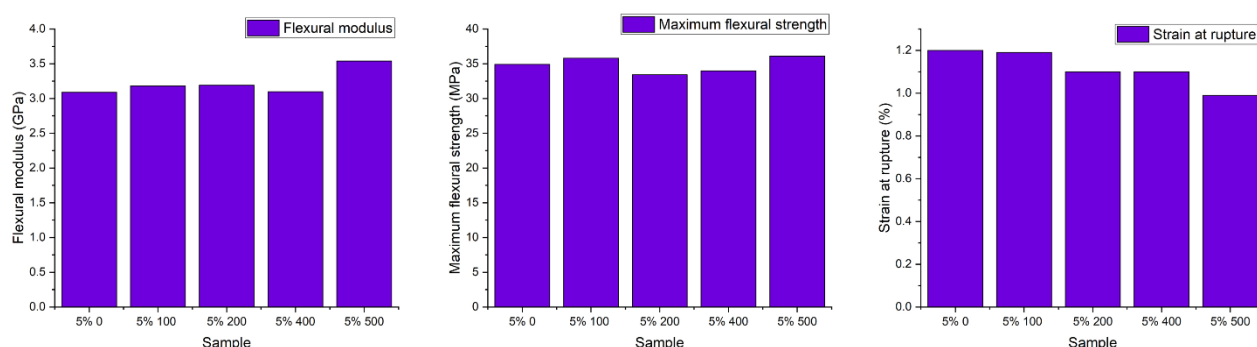


Figure 7: Polyester resin with 5% luffa fibers at 0-500 kGy doses: (a) Flexural modulus, (b) Maximum flexural strength, (c) Strain at rupture

which is 14% higher than that for pure resin value (Figure 7a and Table 4).

In the case of the flexural strength, the values are almost constant, varying from 33.4 to 36.1 MPa, an 8% maximal among them. Thus, minimal resistance is obtained after gamma radiation exposition (Figure 7b).

The strain at rupture decreased values according to the radiation dose increases, from 1.2% to 0.99. The lower was obtained for composites irradiated at 500 kGy, which means 17% less with respect to value for non-irradiated composites (Figure 7c).

Table 4: Results of polyester resin with 5% luffa fibers

Dose (kGy)	Flexural modulus (GPa)	Maximum flexural strength (MPa)	Strain at rupture (%)
0	3.09	35.7	1.20
100	3.18	35.8	1.19
200	3.19	33.4	1.10
400	3.10	34.0	1.10
500	3.54	36.1	0.99



Figure 8. Polyester resin/Luffa fibers composite.

In Figure 8, the cross-sectional area of the specimen is shown, and complete fibers are visible in this area after the rupture. This may indicate a certain lack of adhesion with the matrix, possibly caused by the absence of a pre-treatment on the luffa fibers. Also, Figure 9 displays the set of 'stress vs. strain' graphs of the conducted samples. In it, it can be observed the change in behavior that occurs both when irradiating the polyester resin and when adding luffa fibers.

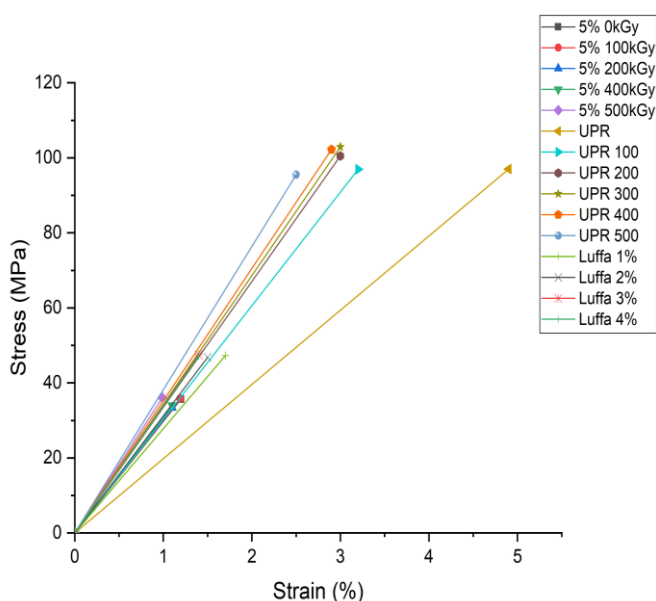


Figure 9. Stress vs strain graphs of the samples.

5. Conclusions

Based on the results, the main conclusions of this study are:

- Gamma radiation is a feasible treatment to solve the problems caused by the loss of styrene monomer in polyester resin and improve the flexural modulus of polyester resin/luffa fiber composites.
- The addition of natural fibers, such as Luffa, is a good option for manufacturing rigid composites due to their availability, low cost, and biodegradability instead of synthetic fibers.

- While at first glance, an increase in material stiffness and a decrease in maximum deformation before failure may seem like an undesirable outcome, a material with these characteristics can find applications in sectors where structural strength, precision, and stability are critical (aerospace industry, instrumentation and measurement, vibration control, materials and equipment testing). However, it is important to carefully consider the specific needs of the application before choosing this type of material, as reduced deformation may not be suitable in all cases.

Abbreviations

UPR: Unsaturated polyester resin

MEKP: Methyl ethyl ketone peroxide

Co. Oct: Cobalt octoate

Acknowledgments:

Special thanks to CONAHCYT for the support through the national scholarship for the "doctoral program in Materials Science offered at UAEMex." Additionally, we extend our gratitude to the Laboratory Services Department at CIATEQ for their technical support in carrying out the project.

References

- Abdel-Hakim, A., Awad, E. H., El-Nemr, K. F., & El-Basheer, T. M. (2021). Impact of gamma radiation and multi-walled carbon nanotubes on the mechanical and acoustical properties of reinforced sisal fiber/polyester resin composites. *Radiation Physics and Chemistry*, 189, 109768. <https://doi.org/10.1016/j.radphyschem.2021.109768>
- Alcañiz-Monge, J., Román-Martínez, M. D. C., & Lillo-Ródenas, M. Á. (2022). Chemical activation of lignocellulosic precursors and residues: what else to consider?. *Molecules*, 27(5), 1630. <https://doi.org/10.3390/molecules27051630>
- Ayma, A., (2017). Effect of Gamma radiation on the properties of jute reinforced polyester matrix composites. *Journal of textile science and engineering* 7 (2): 1-3. <https://doi.org/10.4172/2165-8064.1000294Ddddddddddddddd>
- Bal, K., (2004). Gross morphology and absorption capacity of cell-fibers from the fibrous vascular system of Loofah (*Luffa cylindrica*). *Textile Res. J* 74: 241-247. <https://doi.org/10.1177/004051750407400310>
- Güven, O., Monteiro, S. N., Moura, E. A., & Drelich, J. W. (2016). Re-emerging field of lignocellulosic fiber-polymer composites and ionizing radiation technology in their formulation. *Polymer Reviews*, 56(4), 702-736. <https://doi.org/10.1080/15583724.2016.1176037>
- Haghdan, S., & Smith, G. D. (2015). Natural fiber reinforced polyester composites: A literature review. *Journal of Reinforced Plastics and Composites*, 34(14), 1179-1190. <https://doi.org/10.1177/0731684415588938>
- Hasan, A., Rabbi, M. S., & Billah, M. M. (2022). Making the lignocellulosic fibers chemically compatible for composite: A comprehensive review. *Cleaner Materials*, 4, 100078. <https://doi.org/10.1016/j.clema.2022.100078>
- Hossain, M. T., Hossain, M. S., Kabir, M. S., Ahmed, S., Khan, R. A., & Chowdhury, A. S. (2023). Improvement of mechanical properties of jute-nano cellulose-reinforced unsaturated polyester resin-based composite: Effects of gamma radiation. *Hybrid Advances*, 3, 100068. <https://doi.org/10.1016/j.hybadv.2023.100068>
- Joshi, S. V., Drzal, L. T., Mohanty, A. K., & Arora, S. (2004). Are natural fiber composites environmentally superior to glass fiber reinforced

- composites?. *Composites Part A: Applied science and manufacturing*, 35(3), 371-376.
<https://doi.org/10.1016/j.compositesa.2003.09.016>
- Oboh I., (2009). *Luffa cyllyndrica*- an emerging cash crop. *African journal os agricultural research* 4(8): 684-688. ISBN: 1991-637X
- Qazvini, N. T., & Mohammadi, N. (2005). Dynamic mechanical analysis of segmental relaxation in unsaturated polyester resin networks: Effect of styrene content. *Polymer*, 46(21), 9088-9096.
<https://doi.org/10.1016/j.polymer.2005.06.118>
- Saha, P., Chowdhury, S., Roy, D., Adhikari, B., Kim, J. K., & Thomas, S. (2016). A brief review on the chemical modifications of lignocellulosic fibers for durable engineering composites. *Polymer Bulletin*, 73, 587-620. doi:10.1007/s00289-015-1489-y
- Shen J., 2012. Mechanical properties of *Luffa* sponge. *J. Mech. Behav. Biomed. Mater* 15: 141–152.
<https://doi.org/10.1016/j.jmbbm.2012.07.004>
- Siqueira G., (2010). *Luffa Cylindrica* as a lignocellulosic source of fiber, microfibrillated cellulose, and cellulose nanocrystals. *BioResources* 5 (2): 727-740. ISBN:1930-2126
- Stamatakis, G., Knuutinen, U., Laitinen, K., & Spyros, A. (2010). Analysis and aging of unsaturated polyester resins in contemporary art installations by NMR spectroscopy. *Analytical and bioanalytical chemistry*, 398, 3203-3214.
doi:10.1007/s00216-010-4233-3
- Zhou, Y., Fan, M., Chen, L., & Zhuang, J. (2015). Lignocellulosic fibre mediated rubber composites: An overview. *Composites Part B: Engineering*, 76, 180-191.
<https://doi.org/10.1016/j.compositesb.2015.02.028>