

# Polymer concrete modified by nano-silica particles, waste PET fibers and gamma rays Concreto polimérico modificado con nanopartículas de sílice, fibras de PET de desecho y rayos gamma

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## Abstract

Due to the increasing amount of plastic waste generated year after year, it is necessary to take measures to control this waste and prevent it from accumulating and damaging the environment. To solve these problems, the recycling and reuse of plastic waste have been carried out in the production of composite materials. One of the most novel proposals is the use of high energies, such as gamma rays, to modify and improve the physical and chemical properties of plastic waste. Therefore, in this work, the effects of gamma radiation and nano-silica particles in polymer concretes elaborated with aged polyester resin, silica sand, and waste PET fiber were evaluated. The results show improvements in flexural strength, flexural modulus, and deformation at fracture in the irradiated concretes.

**Keywords:** Polymer concrete, Gamma radiation, Polyester resin, nano-silica, waste PET fibers, mechanical properties.

## Resumen

Debido a la creciente cantidad de residuos plásticos que se generan año tras año, es necesario tomar medidas para controlar estos residuos y evitar que se acumulen y dañen el medio ambiente. Para solucionar estos problemas, se ha llevado a cabo el reciclaje y la reutilización de residuos plásticos en la producción de materiales compuestos. Una de las propuestas más novedosas es el uso de altas energías, como los rayos gamma, para modificar y mejorar las propiedades físicas y químicas de los residuos plásticos. Por ello, en este trabajo se evaluaron los efectos de la radiación gamma y las partículas de nanosílice en hormigones poliméricos elaborados con resina de poliéster envejecida, arena de sílice y residuos de fibra de PET. Los resultados muestran mejoras en la resistencia a la flexión, módulo de flexión y deformación a la fractura en los hormigones irradiados.

**Palabras claves:** Concreto polimérico, radiación gamma, resina poliéster, nano-sílice, fibras de PET de desecho, propiedades mecánicas.

## 1. Introduction

Polymer concretes can be defined as composite materials made by mixing a polymer matrix (of a thermoset or thermoplastic nature) and mineral aggregates (such as silica sand, clay, quartz, or marble). The polymer concrete properties depend on various parameters, such as the polymer type used as a binder, as well as the type, geometry, size, and percentage of mineral aggregates (Elalaoui, 2023; Yelemessov, 2023; Yusof, 2023). Polymer concretes tend to surpass the mechanical properties and corrosion resistance of concrete produced by Portland cement (Figovsky, 2013; Shokrieh, 2015).

Unsaturated polyester resins are employed as thermoset binders for polymer concrete production due to their low cost, high availability, and ease of use (Al-Mufti, 2023; Rasheed, 2023; Shiravi, 2023). These resins are viscous liquid synthetic materials formed by the condensation reaction between a glycol and an unsaturated basic acid. To make unsaturated polyester resins easier to use by reducing their viscosity, they get diluted in a vinyl monomer (usually styrene), which typically represents 25-45% of their mass, and that acts at the same time as a solvent and crosslinking agent (Reuter, 2020; Rubeš, 2024).

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However, when working with UP resins, some conditions related to its processing may influence its final properties. For starters, when curing UP resins at room temperature, the complete setting of its polymer network is never reached due to crosslinking limitations. Moreover, the difficulty of homogeneous mixing of initiators or promoters with UP resins and a lower content in reactive diluent (as in the case of aged polyester resin that's way past its shelf life) further reduces the final degree of crosslinking density achieved (Jurkin, 2006).

It's widely known that incomplete curing in polyester resin has a detrimental effect on its performance to the point of making it more likely to fail when submitted to stress. For this reason, this material is post-cured by exposing it to temperatures at or above the curing temperature for an extended period. This process leads to an improvement in its mechanical properties, a rise in glass transition temperature, reduced residual stress, and reduced tendency to outgas. The post-curing process can also increase crosslinking density and cause backbone dehydration (Ahmed, 2024; Moller, 2020; Silva, 2020).

One of the alternative post-curing processes that have gained some notoriety due to its numerous advantages (including faster reaction times, lower temperature requirements, environmental friendliness, allowance of design adjustments, and being a non-stop and quick process), consists of exposing polymers to different doses of ionizing radiation (Abdel-Rahman, 2018; Eyssa, 2018; da Silva, 2020; Motaleb, 2020).

Gamma radiation can easily create free radicals in polymer networks, causing scission, grafting, and crosslinking reactions that change or improve their physical, mechanical, and chemical properties (Akter, 2020; Rahman, 2019; Tamburrino, 2021). For instance, various studies have focused on exposing polymers such as polyesters, polyurethanes, and other materials to different doses of ionizing radiation to improve their characteristics (Azim, 2020; Babu, 2020; Naikwadi, 2020; Yassene, 2024). Additionally, these studies (Ahmed, 2024; Hamour, 2017; Hossain, 2023; Silva, 2020) point out that gamma radiation can increase bonding between a polymer matrix and a reinforcement in composite materials by increasing the crosslinking structure.

This work focuses on the effect of gamma radiation on the flexural and morphological properties of polymer concrete produced by polyester resin, silica sand, nano-silica, and waste PET fibers. Additionally, this study aims to promote gamma radiation as a viable process for developing composites produced with waste materials, contributing to environmental care.

## 2. Materials and methods

### 2.1. Materials

It was used an aged orthophthalic polyester resin (donated by Concretos Polimericos S.A. de C.V., Mexico). Silica sand

with 80 mesh size (donated by Materiales Gosa, S.A. de C.V., Mexico). Waste PET fibers with a length ranging from 5 to 7 mm and an average diameter of 10  $\mu\text{m}$ , and nano-silica particles supplied from Sigma Aldrich with an average particle size of 200 nm (Code S5505, specific surface 175-225  $\text{m}^2/\text{g}$ , and pH of 3.7-4.3). Additionally, 1.2 wt% methyl ethyl ketone peroxide (MEKP) as the initiator was added to all mixtures.

### 2.2. Types of specimens

In this study, three sets of specimens were made to produce three kinds of polymer concretes:

- Control specimens, produced with resin and silica sand.
- Polymer concretes with 1% wt. of waste PET fibers.
- Polymer concretes with 1% wt. of waste PET fibers, nano-silica particles (0.3 and 0.9 wt%), and irradiated by gamma rays (at 100, 200, and 500 kGy).

It's worth noting that 1% wt. of PET fibers was used because further increasing the content negatively impacted the workability of these composites. Also, these fibers were added to reduce the brittleness and enhance the ductility of concretes.

### 2.3. Preparation of specimens

The steel mold cavities (4x4x16 cm) were coated with wax, then the polymer concrete mixtures were poured into cavities according to EN-196-1 standard, as depicted in Figure 1. Afterward, they were left at room temperature for 48 hours.



Figure 1: Steel mold for specimen preparation.

Control specimens were made by mixing 70 wt% silica sand and 30 wt% polyester resin. The second type of specimens had the same concentration of polyester resin, but 1 wt% of silica sand was substituted by waste PET fibers, which were dispersed in the concrete specimens, as shown in Figure 2.



Figure 2: Mixtures of polyester resin, silica sand and PET fibers.

The third type of specimens was produced by replacing silica sand with nano-silica particles (0.3 and 0.9 wt%), and a single layer of 1 wt% of waste PET fibers. Both materials were uniformly distributed in the mixtures. After each type of specimen was prepared, they were placed at a controlled room temperature for 48 hours without being subjected to any post-curing process.

### 3. Experimental testing

#### 3.1. Flexural tests

The flexural tests were conducted in a Universal testing machine Controls<sup>MR</sup>, according to the ISO-178 standard; located at the Laboratory of Research and Development of Advanced Materials (LIDMA) of the Autonomous University of the State of Mexico.

When testing samples in flexural tests, data was collected by the Controls<sup>MR</sup> software. Consequently, this data was later processed and analyzed in ORIGIN to make the graphs shown in this article.

#### 3.2. Morphological characterization

Waste PET fibers were subjected to 200 kGy and 500 kGy. Afterward, they were dried at a rotatory evaporator for 24 hours, and then their surfaces were analyzed by scanning electron microscopy (SEM) in a JEOL model JSM, located at the Joint Center for Research in Sustainable Chemistry (CCIQS).

### 3.3. Irradiation procedure

Both waste PET fibers and polymer concretes were exposed to gamma radiation doses of 100, 200, and 500 kGy with a 3.5 kGy/h ratio dose. The irradiation was carried out in an Industrial Irradiator JS-6500, located at the National Institute of Nuclear Research (ININ).

## 4. Results and discussion

### 4.1. Polymer concretes with waste PET fibers and nano-silica particles.

Control specimens showed a flexural strength of 24.6 MPa, which was higher when compared with other specimens (Figure 3a). A lower value was achieved for concrete with waste PET fibers, which act as defects and promote crack propagation during flexural tests. However, concretes with waste PET fibers and 0.9 wt.% nanoparticles showed values 4.8% lower than those for control concrete; thus, the nanoparticles enhanced the resistance to high loadings.

A similar pattern was seen in the flexural modulus (Figure 3b) since control specimens showed a flexural modulus of 1.08 GPa, which was significantly higher when compared with those containing waste PET fibers and nano-silica particles.

The strain at break shows a higher value for polymer concretes with 0.9 wt.% of nano-silica particles compared to control concrete, which was 6% higher (Figure 3c). However, the values for concrete with PET fibers or 0.3 wt.% nano-silica were lower than those for control concrete.



Figure 4: Control specimens (a), and concrete with PET fibers (b), after flexural tests.

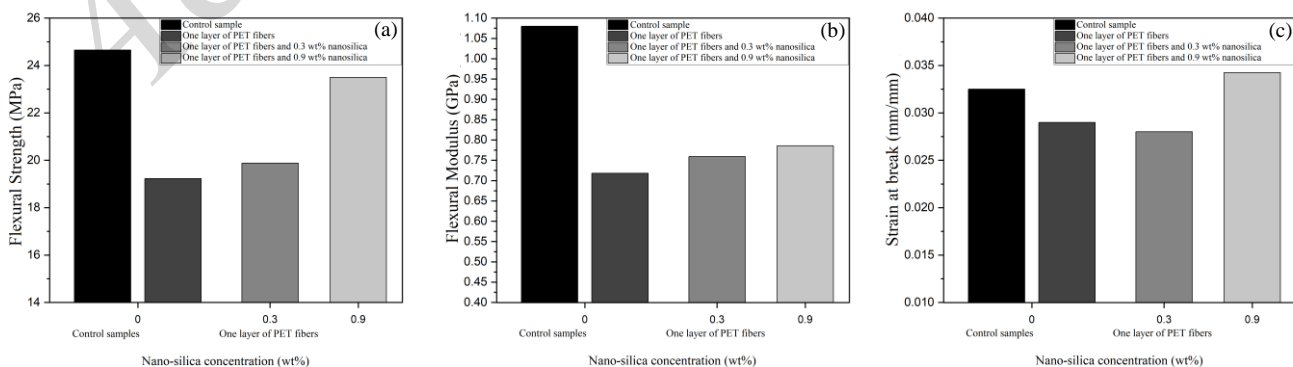


Figure 3: Polymer concretes with waste PET fibers and nano-silica particles.



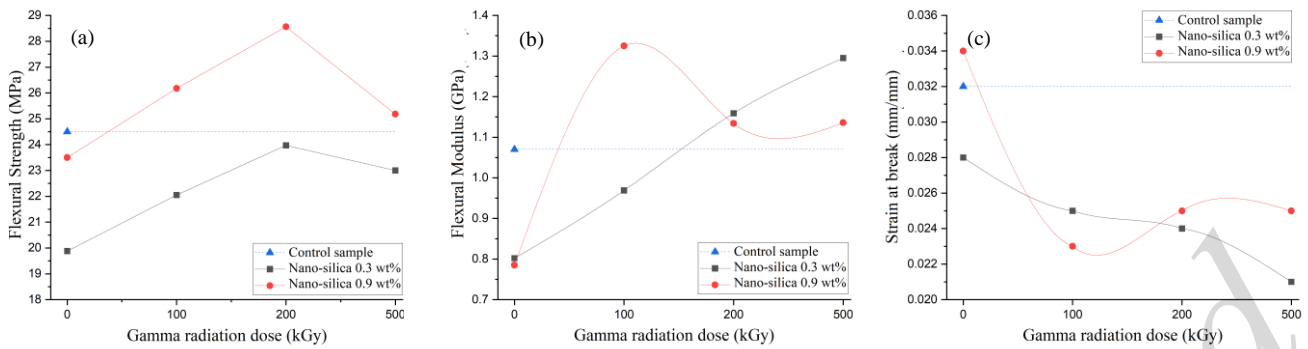


Figure 5: Polymer concretes with waste PET fibers and nano-silica particles exposed to gamma radiation.

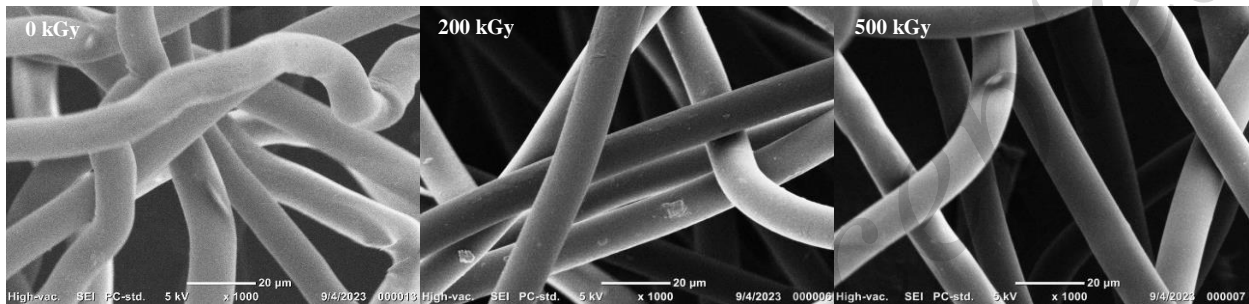


Figure 6: Waste PET fibers exposed to gamma radiation.

Control specimens exhibited a continuous fracture after the flexural test (Figure 4a). However, polymer concretes with PET fibers exhibited a mitigation on the crack propagation after flexural tests (4b).

#### 4.2. Polymer concretes with waste PET fibers and nano-silica particles, exposed to gamma radiation.

After exposing to gamma radiation dose, the specimens with waste PET fibers, 0.9 wt% of nano-silica, and irradiated at 200 kGy showed a drastic increase in the flexural strength compared to control concrete (Figure 5a). However, specimens with 0.3% nano-silica had lower values than that for control concrete. This improvement arises from gamma radiation exposure, which forces free styrene content and unreacted polyester double bonds still present within the polymer network to take part in the copolymerization of styrene with polyester, as well as homo-polymerization of styrene and polyester reactions that reduce polymer chains mobility and increase crosslinking density. It's worth mentioning that augmenting the dose of gamma radiation used normally leads to a higher degree of crosslinking density on polyester resin. However, at high radiation doses, chain scission becomes more prevalent than the crosslinking process, causing a decrease in mechanical properties (Ashfaq, 2020; Gao, 2019; Singh, 2022).

In the case of the flexural modulus values, these tended to increase after exposure to gamma rays (Figure 5b), which produced an increase in the density through cross-linking of polymer chains. It was noticeable that high flexural modulus was achieved either using a high radiation dose and low nano-silica concentration or using a low gamma dose and high nano-silica concentration.

Furthermore, all concretes exposed to gamma rays exhibited lower strain at break compared to that for control concrete (Figure 5c). The increase of cross-linking of polymer chains in polyester resin restricts their mobility and modifies the interface between nano-silica particles and resin, thus the strain is reduced.

#### 4.3. Morphological properties

Non-irradiated waste PET fibers exhibit a smooth surface, whereas irradiated fibers show small particles and wrinkles (Figure 6), resulting from polymer chain breakage and surface degradation. However, this process creates contact points that enhance interaction with polyester resin.

### 5. Conclusions

Little information exists regarding ionizing radiation as a post-curing method of polymer concretes and its use for enhancing the properties of composites produced with waste materials. For this reason, this study centers on examining the effect of gamma radiation at different doses on the flexural and morphological properties of polymer concretes produced with silica sand, waste materials (aged polyester resin and PET fibers) and, reinforced with nano-silica.

Experimental results show that PET fibers can decrease flexural strength, flexural modulus, and strain at break when added as a layer in polymer concretes; however, its presence can also mitigate the propagation of cracks that occur after the specimens fail. On the other hand, by the addition of nano-silica particles in its composition, polymer concretes made with waste PET fibers show superior flexural strength, flexural modulus, and strain at break.

Additionally, a significant increase in flexural strength and flexural modulus occurred for irradiated concrete. The improvement was due to an increase in the cross-linking of polymer chains. Additionally, gamma radiation modifies the surface of waste PET fibers, enhancing their interaction with polyester resin and thereby improving the ability of these composites to distribute applied loads.

Future work will be considered to study the effects of using different sizes of nano-silica in polymer concretes to further analyze its effectiveness as a reinforcement in composite materials submitted to gamma radiation. Additionally, the effect of gamma radiation on the rheological properties of polyester resin with nano-silica needs further investigation.

## References

- Abdel-Rahman, H.A., Younes, M.M., & Yassene, A.A. (2018). Physico-mechanical properties of gamma-irradiated clay/polyester nanocomposites. *Polymer Composites*, 39(10), 3666-3675. <https://doi.org/10.1002/pc.24395>
- Ahmed, M. S., Haque, M. M., Khan, R. A., & Miah, M. A. H. (2024). A Review on Properties of Natural Fiber Reinforced Polymer Composites: Effect of Gamma Radiation and Nano particles. *International Journal of Engineering Materials and Manufacture*, 9(1), 15-29. <https://doi.org/10.26776/ijemm.09.01.2024.02>
- Akter, Nasrin Das, Subrata Grammatikos, Sotirios Saha, Joykrishna & Khan, Mubarak. (2020). Development of sustainable jute geotextiles by bitumen emulsion and polyester resin: Effect of gamma radiation. *Journal of Engineered Fibers and Fabrics*, 15. <https://doi.org/10.1177/1558925020957969>.
- Al-Mufti, S. M., Almontasser, A., & Rizvi, S. J. (2023). Unsaturated Polyester Resin Filled with Cementitious Materials: A Comprehensive Study of Filler Loading Impact on Mechanical Properties, Microstructure, and Water Absorption. *ACS omega*, 8(23), 20389-20403. <https://doi.org/10.1021/acsomega.3c00353>
- Ashfaq, A., Clochard, M.C., Coqueret, X., Dispenza, C., Driscoll, M.S., Ulański, P., & Al-Sheikhly, M. (2020). Polymerization reactions and modifications of polymers by ionizing radiation. *Polymers*, 12(12), 2877. <https://doi.org/10.3390/polym12122877>
- Azim, A. Y. M. A., Alimuzzaman, S., & Sarker, F. (2022). Optimizing the fabric architecture and effect of  $\gamma$ -radiation on the mechanical properties of jute fiber reinforced polyester composites. *ACS omega*, 7(12), 10127-10136. <https://doi.org/10.1021/acsomega.1c06241>
- Babu, M. S., Sarathi, R., Vasa, N. J., & Imai, T. (2020). Investigation on space charge and charge trap characteristics of gamma-irradiated epoxy micro-nano composites. *High Volt*, 5(2), 191-201. <https://doi.org/10.1049/hve.2019.0188>
- da Silva, A. O., de Castro Monsorens, K. G., Oliveira, S. D. S. A., Weber, R. P., Monteiro, S. N., & de Carvalho Vital, H. (2020). Influence of gamma and ultraviolet radiation on the mechanical behavior of a hybrid polyester composite reinforced with curaua mat and aramid fabric. *Journal of Materials Research and Technology*, 9(1), 394-403. <https://doi.org/10.1016/j.jmrt.2019.10.068>
- Elalouai, O. (2023). Effect of Short Fibers on Fracture Properties of Epoxy-Based Polymer Concrete Exposed to High Temperatures. *Polymers*, 15(5), 1078. <https://doi.org/10.3390/polym15051078>
- Eyssa, H.M., Abulyazied, D.E., Abdulrahman, M., & Youssef, H.A. (2018). Mechanical and physical properties of nanosilica/nitrile butadiene rubber composites cured by gamma irradiation. *Egyptian Journal of petroleum*, 27(3), 383-392. <https://doi.org/10.1016/j.ejpe.2017.06.004>
- Figovsky, O., & Beilin, D. (2013). *Advanced polymer concretes and compounds*. CRC Press. pp. 1-21. <https://doi.org/10.1201/b16237>
- Gao, Y., Zhang, H., Huang, M., & Lai, F. (2019). Unsaturated polyester resin concrete: A review. *Construction and Building Materials*, 228, 116709. <https://doi.org/10.1016/j.conbuildmat.2019.116709>
- Hamour, N. Amar, Boukerrou & Hocine. (2017). Effect of gamma irradiation aging on mechanical and thermal properties of alfa fiber-reinforced polypropylene composites: Role of alfa fiber surface treatments. *Journal of Thermoplastic Composite Materials*, 31, 1-18. <https://doi.org/10.1177/0892705717714831>.
- 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>
- Jurkin, T., & Pucić, I. (2006). Post-irradiation crosslinking of partially cured unsaturated polyester resin. *Radiation Physics and Chemistry*, 75(9), 1060-1068. <https://doi.org/10.1016/j.radphyschem.2006.04.001>
- Moller, J. C., Berry, R. J., & Foster, H. A. (2020). On the nature of epoxy resin post-curing. *Polymers*, 12(2), 466. <https://doi.org/10.3390/polym12020466>
- Motaleb, K.A., Milasius, R., & Ahad, A. (2020). Influence of gamma radiation on mechanical properties of jute fabric-reinforced polymer composites. *Fibers*, 8(9), 58. <https://doi.org/10.3390/fib8090058>
- Naikwadi, A. T., Sharma, B. K., Bhatt, K. D., & Mahanwar, P. A. (2022). Gamma radiation processed polymeric materials for high performance applications: a review. *Frontiers in Chemistry*, 10, 837111. <https://doi.org/10.3389/fchem.2022.837111>
- Rahman, M. T. (2019). Fe2O3 nanoparticles dispersed unsaturated polyester resin-based nanocomposites: effect of gamma radiation on mechanical properties. *Radiation Effects and Defects in Solids*, 174 (5-6), 480-493. <https://doi.org/10.1080/10420150.2019.1606809>
- Rasheed, A., Sadiq, S., & Shaaban, A. (2023). Effect of varied waste concrete ratios on the mechanical properties of polymer concrete. *Open Engineering*, 13(1), 20220468. <https://doi.org/10.1515/eng-2022-0468>
- Reuter, J., Greiner, L., Kukla, P., & Döring, M. (2020). Efficient flame-retardant interplay of unsaturated polyester resin formulations based on ammonium polyphosphate. *Polymer degradation and stability*, 178, 109134. <https://doi.org/10.1016/j.polydegradstab.2020.109134>
- Rubeš, D., Vinklárček, J., Podzimek, Š., & Honzický, J. (2024). Bio-based unsaturated polyester resin from post-consumer PET. *RSC advances*, 14(12), 8536-8547. <https://doi.org/10.1039/D3RA08500G>
- Shiravi, M. M., & Eftekhari, M. R. (2023). Analyzing the bond strength between lightweight concrete and lightweight polyester-polymer concrete. *Innovative Infrastructure Solutions*, 8(1), 52. <https://doi.org/10.1007/s41062-022-01012-6>
- Shokrieh, M. (2015). A novel polymer concrete made from fine silica sand and polyester. *Mechanics of Composite Materials*, 51, 571-580. <https://doi.org/10.1007/s11029-015-9528-1>
- Silva, M. P., Santos, P., Parente, J. M., Valvez, S., Reis, P. N., & Piedade, A. P. (2020). Effect of post-cure on the static and viscoelastic properties of a polyester resin. *Polymers*, 12(9), 1927. <https://doi.org/10.3390/polym12091927>
- Singh, A. K., Adhikari, R., & Susan, M. A. B. H. (2022). Modification of polymers with gamma radiation for various high-performance applications. *Frontiers in Chemistry*, 10, 1042056. <https://doi.org/10.3389/fchem.2022.1042056>
- Tamburrino, F., Barone, S., Paoli, A., & Razonale, A. V. (2021). Post-processing treatments to enhance additively manufactured polymeric parts: A review. *Virtual and Physical Prototyping*, 16(2), 221-254. <https://doi.org/10.1080/17452759.2021.1917039>
- Yassene, A. A., Awad, E. H., & Hegazy, A. A. (2024). The effect of gamma rays and stearic acid on calcium carbonate and its impact on the properties of epoxy-based composites. *Radiochimica Acta*, (0). <https://doi.org/10.1515/ract-2023-0266>
- Yelemessov, K., Sabirova, L. B., Martushev, N. V., Malozyomov, B. V., Bakhmagambetova, G. B., & Atanova, O. V. (2023). Modeling and Model Verification of the Stress-Strain State of Reinforced Polymer Concrete. *Materials*, 16(9), 3494. <https://doi.org/10.3390/ma16093494>
- Yusuf, M. Y. M., & Shahidan, S. (2023). A Comprehensive Review on Physical and Mechanical Properties of Polymer as Concrete Material. *Recent Trends in Civil Engineering and Built Environment*, 4(3), 70-83. <https://doi.org/10.30880/rctcebe.2023.04.03.008>