ISSN 1061-8309, Russian Journal of Nondestructive Testing, 2024, Vol. 60, No. 6, pp. 692–698. © Pleiades Publishing, Ltd., 2024.

X-RAY METHODS

Characterization of Euphorbia Tortilis Cactus Concrete Specimen by 3D X-ray Tomography

K. M. Gopalakrishnan[a], R. Mohanraj[b,*], S. Southamirajan[c], and S. Ramkumar[d]

[a] *Civil Engineering, Erode Sengunthar Engineering College, Erode, Tamilnadu, 638057 India*

[b] *Civil Engineering, SRM University, Delhi-NCR, Sonepat, Haryana, 131028 India*

[c] *Civil Engineering, Kongunadu College of Engineering and Technology, Trichy, Tamilnadu, 621215 India*

[d] *Civil Engineering, K. Ramakrishnan College of Technology, Trichy, Tamilnadu, 621112 India*

** e-mail: rsrirammohan@srmuniversity.ac.in*

Received May 7, 2024; revised May 28, 2024; accepted June 7, 2024

Abstract—This study investigates the enhancement of Euphorbia Tortilis Cactus (ETC) infused concrete, focusing on its microstructural characteristics and mechanical properties. ETC concrete, known for superior tensile ductility and durability, was characterized using advanced 3D X-ray tomography and energy dispersive X-ray spectroscopy (EDX) analysis. Specimens with a 9% ETC extract mix underwent high-resolution Micro Computed Tomography scanning, revealing an average porosity of 3.3% and providing detailed insights into internal features like pores, fibers, and aggregates. EDX mapping identified key elements, including Si, O, Mg, and Ca, highlighting calcium carbonate and brucite formations. This research addresses gaps in understanding ETC concrete's microstructure and mechanical behavior, using non-destructive imaging and chemical analysis to offer comprehensive insights. The findings suggest that ETC concrete can be optimized for enhanced performance in sustainable construction, highlighting its potential for applications requiring high durability and ductility. The study's novelty lies in its application of advanced imaging and analysis techniques to optimize ETC concrete mix designs, assess durability, and develop strategies for mitigating structural issues, thus providing valuable insights into the material's properties and behavior.

Keywords: X-ray, tomography, EDX, cactus, concrete, MicroCT

DOI: 10.1134/S1061830924601892

1. INTRODUCTION

Euphorbia Tortilis Cactus (ETC) concrete has garnered significant attention in recent years due to its potential applications and exceptional mechanical properties in various fields of construction and infrastructure [1–3]. These advanced cement-based materials are engineered to exhibit improved tensile ductility, enhanced durability, and superior performance compared to traditional concrete [4–6]. The unique properties of ETC stem from its microstructure, which is carefully engineered to incorporate various additives and fibers to enhance its mechanical behavior [7, 8]. Unlike conventional concrete, which is brittle and prone to cracking under tensile stresses, ETC exhibits remarkable tensile strain capacity and crack control mechanisms, making it suitable for a wide range of applications, including bridge decks, earthquake-resistant structures, marine infrastructure, and repair materials for aging structures [9, 10].

The key to the superior performance of ETC lies in its microstructural design, which involves optimizing the distribution of cementitious materials, fibers, and other additives at the microscopic level [11, 12]. Understanding the complex microstructure of ETC is essential for optimizing its properties, predicting its behavior under different loading conditions, and ensuring its long-term durability [13]. However, characterizing the microstructure of ETC poses significant challenges due to its heterogeneous nature and the presence of various internal elements, such as fibers, pores, and hydration products [14]. Conventional techniques for microstructural analysis, such as optical microscopy and scanning electron microscopy (SEM), provide valuable information but are limited in their ability to visualize the three-dimensional (3D) structure of ETC comprehensively [15, 16]. To overcome these limitations, advanced imaging techniques such as X-ray tomography and MicroCT scanning have emerged as powerful tools for nondestructive 3D imaging and analysis of materials [17–19].

X-ray tomography, in particular, offers unique capabilities for visualizing the internal structure of ETC specimens in three dimensions with high resolution [20]. By utilizing X-rays to penetrate the sample and capture projection images from multiple angles, X-ray tomography enables the reconstruction of a detailed 3D volume representing the internal microstructure of ETC [21]. This non-destructive imaging technique provides valuable insights into the distribution and morphology of internal elements within the material, allowing researchers to analyze the spatial arrangement of fibers, detect defects, and quantify porosity with unprecedented accuracy. MicroCT scanning, a subtype of X-ray tomography, further enhances imaging capabilities by offering high-resolution imaging of ETC specimens at the micrometer scale. MicroCT scans provide detailed insights into the internal microstructure of ETC, allowing for the visualization and quantification of pores, fibers, aggregates, and other internal elements. Additionally, microCT scanning offers advantages such as rapid scanning time, efficient analysis of large volumes of material, and the ability to visualize macroscopic features such as voids and cracks [22]. These advantages make MicroCT scanning an invaluable tool for researchers and engineers seeking to understand the microstructural properties of ETC and optimize its performance for various applications.

This study aims to explore and enhance the properties of ETC infused concrete, particularly focusing on its mechanical performance and sustainability. The primary objectives are to investigate the internal structure of ETC concrete using advanced 3D X-ray tomography and EDX analysis, quantify its microstructural features such as porosity and fiber distribution. Previous studies have not fully addressed the challenges of characterizing the heterogeneous microstructure of ETC concrete, especially in terms of nondestructive imaging and accurate chemical composition analysis. The research gap lies in the lack of comprehensive understanding of the internal microstructural behavior and the distribution of constituents within ETC concrete. The novelty of this work is highlighted by the integration of high-resolution MicroCT scanning and detailed EDX mapping, which provide unprecedented insights into the material's internal features and chemical composition. This approach not only advances the characterization techniques for ETC concrete but also offers a deeper understanding of its microstructural properties, paving the way for optimizing mix designs and enhancing its application in sustainable construction.

2. MATERIALS AND METHODS

ETC specimens were prepared according to standard procedures, incorporating cementitious materials, aggregates, and extract of cactus (Euphorbia tortilis). Concrete mixed with the optimum level of 9% Euphorbia tortilis cactus extract (ETC9). A high-resolution MicroCT system with a flat-panel detector and a micro-focus X-ray source was employed for imaging [23, 24]. Specimens were mounted on a rotational stage to capture X-ray projection images from multiple angles [25]. Reconstruction of 3D volumes was performed using specialized software, allowing visualization and quantification of internal microstructural features such as pores, fibers, and aggregates [26].

3. EXPERIMENTAL

The experimental setup consists of a high-resolution X-ray tomography system equipped with a flat-panel detector and a micro-focus X-ray source. ETC concrete specimens of different sizes and compositions were prepared, representing typical mixes used in construction. Samples were cured under various conditions to simulate real-world scenarios, including different water-to-cement ratios, curing temperatures, and supplementary cementitious materials [3]. The ETC concrete specimens were subjected to X-ray tomography analysis, with multiple projection images acquired over a range of rotation angles [27]. Reconstruction algorithms, such as filtered back projection or iterative methods, were employed to reconstruct the 3D volumes from the acquired projection data. The reconstructed volumes were visualized and analyzed using specialized software tools to extract quantitative parameters.

4. RESULT AND DISCUSSION

4.1. 3D X-Ray Tomography Analysis

The X-ray tomography apparatus produces a three-dimensional image of the material that emphasises the presence of internal constituents and their distribution pattern. Predicting the orientation of the chemicals and the failure pattern is made easier by the image. Figure 1 shows the typical perspective view of the image with the optimal mix ETC9. MicroCT scanning is a powerful imaging technique that provides detailed insights into the internal structure of materials, including ETC concrete specimens. At a resolution of 100 μm, MicroCT scans offer high-resolution imaging capabilities, allowing for thorough exam-

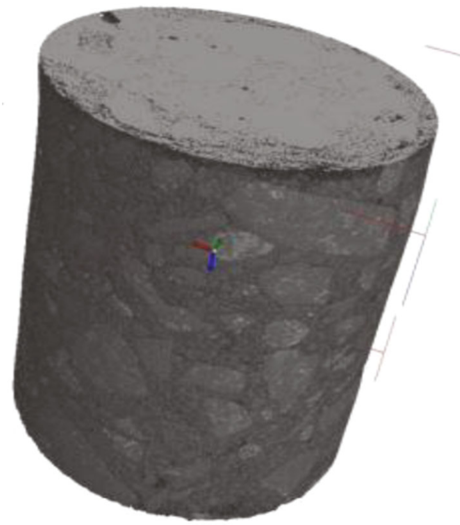

Fig. 1. Perspective view.

ination of the sample's microstructure. One of the notable advantages of MicroCT is its efficiency, as an entire sample can be scanned in a relatively short time, typically less than 5 min. This rapid scanning process enables researchers to analyze large volumes of material quickly, making it particularly useful for applications where time is of the essence. In Fig. 1, we can observe an example of a MicroCT scan of a ETC concrete cylinder. The voids within the ETC concrete are removed, and their 3D visualization is depicted in blue coloration to the right of the exterior surface view. This visualization technique provides valuable insights into the distribution and morphology of pores within the ETC concrete specimen. In the example shown, the average porosity is calculated to be 3.3%. This quantitative information helps in understanding the material's overall quality and performance characteristics.

The fast MicroCT method is especially advantageous when the focus is on identifying and analyzing the largest pores within the material. By swiftly capturing images at a high resolution, it can effectively investigate macroscopic features such as voids and cracks. The smallest detectable pore diameter is typically double the scan resolution, which means that in this case, the smallest detected pore diameter is 200 μm. This limitation should be considered when interpreting the results, especially for applications requiring precise characterization of fine-scale features. Furthermore, MicroCT scanning allows for the generation of data histograms (Fig. 3), which illustrate the separation between air and material within the sample. Figure 3 provides valuable quantitative data regarding the distribution of pore sizes and the variation between scans of different ETC concrete samples. It is essential to recognize that each scan may require its threshold value to accurately distinguish between air and material phases. Adjusting the threshold value ensures the reliability and reproducibility of the analysis, particularly when comparing results from different samples or experimental conditions. Overall, MicroCT scanning offers a non-destructive and efficient means of characterizing the microstructure of ETC concrete specimens. By providing detailed 3D visualization and quantitative data on pore characteristics, MicroCT enables researchers to gain valuable insights into the material's properties and behavior. This information is essential for optimizing ETC concrete mix designs, assessing durability, and developing strategies for mitigating potential structural issues. As technology continues to advance, MicroCT scanning is likely to become an increasingly indispensable tool in the field of materials science and engineering.

The thresholding operation in MicroCT scanning serves as a critical step in distinguishing between air and material phases within the scanned sample. However, it is important to acknowledge that this operation is subject to manual human interface, which introduces an inherent error margin based on human perception of the edge of the void region. This human involvement can lead to inconsistencies and inaccuracies in the determination of the threshold value, particularly when defining the boundary between voids and solid material. Furthermore, image artifacts can further complicate the thresholding process and contribute to errors in the analysis. For example, beam hardening artifacts may occur, causing small voids near the edge of the sample to appear brighter than identical voids in the middle part of the sample. This discrepancy arises due to gradual intensity variations across the sample, which can distort the perception of voids and lead to misinterpretations in the thresholding operation. Figure 2 illustrates the 2D

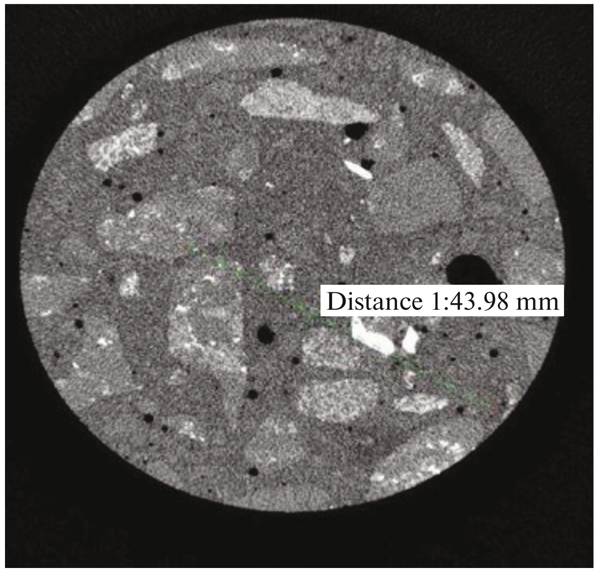

Fig. 2. 2D view.

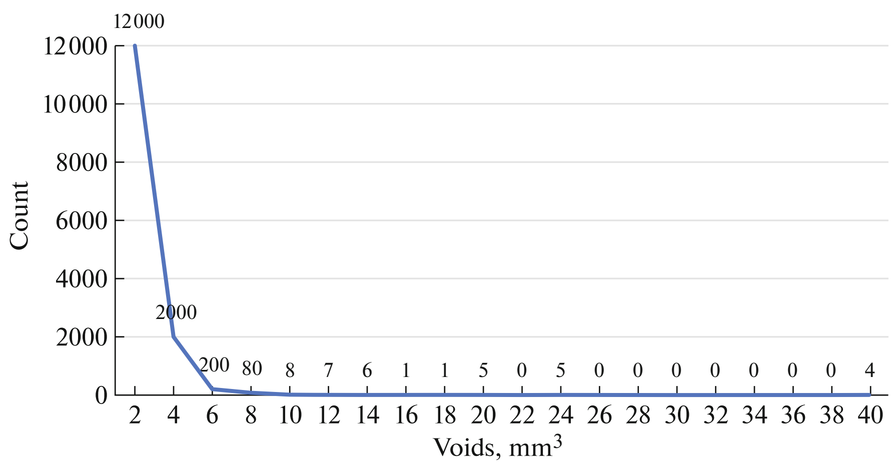

Fig. 3. Histogram of the void size distribution of the ETC concrete sample.

analysis of slices from top to bottom through the sample, revealing significant variation in porosity levels within the specimen. The porosity varies roughly from 2.5 to 5%, indicating substantial heterogeneity in the material's microstructure. While human measurement error would typically fall within this range using conventional thresholding methods, the 2D porosity analysis depicted in Fig. 2 eliminates human error as the threshold remains consistent across all slices. This approach ensures greater accuracy and reliability in porosity assessment, as it minimizes the influence of subjective human perception on the thresholding process [1]. By employing consistent thresholding across all slices, researchers can mitigate the impact of human error and obtain more reliable quantitative data on porosity variation within the sample. This methodology enhances the reproducibility and robustness of MicroCT analysis, enabling more accurate characterization of material microstructures and facilitating informed decision-making in various scientific and engineering applications.

4.2. EDX Analysis

The detailed chemical composition of precipitates was analyzed through SEM images accompanied by EDX mapping, as depicted in Fig. 4. These images provided insights into the concentration distributions of O, Si, Mg, and Ca, corresponding to specific regions highlighted in Fig. 4. Notably, the presence of

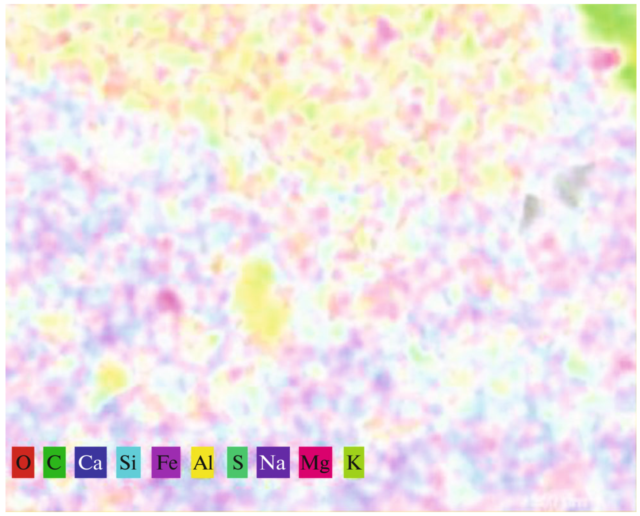

Fig. 4. Group mapping by spectrometer.

a high concentration of C in certain regions was attributed to the carbon deposition and cactus extract (polysaccharides). Sealing deposits exhibited a predominant composition of Ca, C, and O, suggesting the prevalence of calcium carbonate. This composition was consistently observed in the sealing deposits on the specimen's surface end and in certain regions of the specimen's surface. However, such deposits were notably absent on the macro-fracture surface within the specimen's interior, indicating localized deposition patterns. In contrast, precipitates containing Mg were found to cover the entire surface of the specimen, including macro-fracture surfaces. The presence of O alongside Mg suggested the formation of brucite or dolomite. The absence of dissolved carbonate ions implied brucite formation, supported by previous research findings. Considering the age of the ETC specimen, approximately 2 year, the primary mechanism for sealing macro-fractures near the specimen's end under the tested conditions was attributed to aragonite deposition over a layer of brucite. This finding suggests that fractures with apertures smaller than 0.1 mm may be effectively sealed by brucite alone [3]. The SEM observations and chemical analyses provide valuable insights into the composition and formation mechanisms of precipitates, shedding light on the sealing behavior of macro-fractures in the specimen under investigation.

5. CONCLUSIONS

(i) MicroCT scanning emerges as a powerful technique for the non-destructive characterization of ETC concrete microstructure. The study demonstrates the effectiveness of MicroCT in visualizing and quantifying pore distribution within the specimen, offering valuable insights into its properties and behavior. Despite challenges such as manual thresholding and image artifacts, MicroCT remains a valuable tool for material analysis and optimization. Moving forward, further advancements in MicroCT technology and data analysis techniques are expected to enhance its capabilities and expand its applications in materials science and engineering. Overall, this study underscores the importance of MicroCT scanning in advancing our understanding of ETC concrete microstructure and guiding efforts toward improving material performance and durability.

(ii) SEM analysis revealed calcium carbonate predominance in sealing deposits near the specimen's surface end, while brucite or dolomite covered the entire surface, including macro-fractures. Limited dissolved carbonate ions suggested brucite formation. The age of the specimen indicated aragonite deposition over brucite, effectively sealing macro-fractures near the specimen's end. Fractures with apertures smaller than 0.1 mm may be sealed solely by brucite.

FUNDING

This work was supported by ongoing institutional funding. No additional grants to carry out or direct this particular research were obtained.

CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

REFERENCES

1. Du Plessis, A. et al., Simple and fast porosity analysis of concrete using X-ray computed tomography, *Mater. Struct.,* 2016, vol. 49, pp. 553–562.
2. Shanmugavel, D., et al., Interaction of a viscous biopolymer from cactus extract with cement paste to produce sustainable concrete, *Constr. Build. Mater.,* 2020, vol. 257, p. 119585.
3. Mishnaevskiy, L.L., Automatic voxel-based generation of 3D microstructural FE models and its application to the damage analysis of composites, *Mater. Sci. Eng. A,* 2005, vol. 407, pp. 11–23.
4. Balázs, G.L., et al., Evaluation of concrete elements with X-ray computed tomography, *J. Mater. Civ. Eng.,* 2018, vol. 30, no. 9.
5. Woldemariam, A.M., Oyawa, W.O., and Abuodha, S.O., The use of plant extract as shrinkage reducing admixture (SRA) to reduce early age shrinkage and cracking on cement mortar, *Int. J. Innov. Sci. Res.,* 2015, vol. 13, pp. 136–144.
6. Vaidevi, C., Compline, D.V., Vishnuram, R., Arun, A., Musaffer, M.M., and Sharen, S.S., Study on properties of cactus in concrete-cactus concrete, *ECS Trans.,* 2022, vol. 107, p. 19041. https://doi.org/10.1149/10701.19041ecst
7. Shanmugavel, D., Selvaraj, T., Ramadoss, R., and Raneri, S., Interaction of a viscous biopolymer from cactus extract with cement paste to produce sustainable concrete, *Constr. Build. Mater.,* 2020, vol. 257, p. 119585. https://doi.org/10.1016/j.conbuildmat.2020.119585
8. Mohanraj, R. and Senthilkumar, S., *Experimental studies on concrete prepared with extract of euphorbia tortilis cactus as a natural*, Anna University, India, 2023. http://hdl.handle.net/10603/519286.
9. Rouhou, M.C., Abdelmoumen, S., Thomas, S., Attia, H., and Ghorbel, D., Use of green chemistry methods in the extraction of dietary fibers from cactus rackets (Opuntia ficus indica): Structural and microstructural studies, *Int. J. Biol. Macromol.,* 2018, vol. 116, pp. 901–910. https://doi.org/10.1016/j.ijbiomac.2018.05.090
10. Ramírez-Arellanes, S., Cano-Barrita, P.D.J., Julián-Caballero, F., and Gómez-Yañez, C., Concrete durability properties and microstructural analysis of cement pastes with nopal cactus mucilage as a natural additive, *Mater. Constr.,* 2012, vol. 62, pp. 327–341.
11. Peschard, A., Govin, A., Grosseau, P., Guilhot, B., and Guyonnet, R., Effect of polysaccharides on the hydration of cement paste at early ages, *Cem. Concr. Res.,* 2004, vol. 34, pp. 2153–2158. https://doi.org/10.1016/j.cemconres.2004.04.001
12. Pattusamy, L., Rajendran, M., Shanmugamoorthy, S., and Ravikumar, K., Confinement effectiveness of 2900psi concrete using the extract of Euphorbia tortilis cactus as a natural additive, *Matéria (Rio J.),* 2023, vol. 28. https://doi.org/10.1590/1517-7076-RMAT-2022-0233
13. Martínez-Molina, W., Torres-Acosta, A.A., Celis-Mendoza, C.E., and Alonso-Guzman, E., Physical properties of cement-based paste and mortar with dehydrated cacti additions, *Int. J. Archit. Herit.,* 2015, vol. 9, pp. 443–452. https://doi.org/10.1080/15583058.2013.800919
14. Loganathan, P., Mohanraj, R., Senthilkumar, S., and Yuvaraj, K., Mechanical performance of ETC RC beam with U-framed AFRP laminates under a static load condition, *Rev. Constr.,* 2022, vol. 21, pp. 678–691. https://doi.org/10.7764/RDLC.21.3.678
15. Mohanraj, R., Senthilkumar, S., Goel, P., and Bharti, R., A state-of-the-art review of Euphorbia Tortilis cactus as a bio-additive for sustainable construction materials, *Mater. Today Proc.,* 2023. https://doi.org/10.1016/j.matpr.2023.03.762
16. Hazarika, A., Hazarika, I., Gogoi, M., Bora, S.S., Borah, R.R., Goutam, P.J., and Saikia, N., Use of a plant-based polymeric material as a low-cost chemical admixture in cement mortar and concrete preparations, *J. Build. Eng.,* 2018, vol. 15, pp. 194–202. https://doi.org/10.1016/j.jobe.2017.11.017
17. Hernández, E.F., Cano-Barrita, P.D.J., and Torres-Acosta, A.A., Influence of cactus mucilage and marine brown algae extract on the compressive strength and durability of concrete, *Mater. Constr.,* 2016, vol. 66, p. e074. https://doi.org/10.3989/mc.2016.07514
18. Bouakba, M., Bezazi, A., Boba, K., Scarpa, F., and Bellamy, S., Cactus fibre/polyester biocomposites: Manufacturing, quasi-static mechanical and fatigue characterisation, *Compos. Sci. Technol.,* 2013, vol. 74, pp. 150–159. https://doi.org/10.1016/j.compscitech.2012.10.009

19. Chandra, S., Eklund, L., and Villarreal, R.R., Use of cactus in mortars and concrete, *Cem. Concr. Res.,* 1998, vol. 28, pp. 41–51.
https://doi.org/10.1016/S0008-8846(97)00254-8
20. Shanmugasundaram, S., Mohanraj, R., Senthilkumar, S., and Padmapoorani, P., Torsional performance of reinforced concrete beam with carbon fiber and aramid fiber laminates, *Rev. Constr.,* 2022, vol. 21, pp. 329–337.
https://doi.org/10.7764/RDLC.21.2.329
21. Cárdenas, A., Goycoolea, F.M., and Rinaudo, M., On the gelling behaviour of 'nopal' (Opuntia ficus indica) low methoxyl pectin, *Carbohydr. Polym.,* 2008, vol. 73, pp. 212–222.
https://doi.org/10.1016/j.carbpol.2007.11.017
22. Mohanraj, R., Senthilkumar, S., Amutham, B., and Krishnasamy, R., Concrete strength: Rubber crumbs & steel slag mix – Python analysis, *Comput. Eng. Phys. Model.,* 2024, vol. 7, no. 2.
https://doi.org/10.22115/cepm.2024.450919.1296
23. Ravi, R., Selvaraj, T., and Sekar, S.K., Characterization of hydraulic lime mortar containing Opuntia ficus-indica as a bio-admixture for restoration applications, *Int. J. Archit. Herit.,* 2016, vol. 10, pp. 714–725.
https://doi.org/10.1080/15583058.2015.1109735
24. Mohanraj, R., Senthilkumar, S., and Padmapoorani, P., Mechanical properties of RC beams with AFRP sheets under a sustained load, *Mater. Technol.,* 2022, vol. 56, pp. 365–372.
https://doi.org/10.17222/mit.2022.481
25. Manikandan, A.T. and Padmavathi, A., An experimental investigation on improvement of concrete serviceability by using bacterial mineral precipitation, *Int. J. Res. Sci. Innov.,* 2015, vol. 2.
26. Mohanraj, R. and Vidhya, K., Evaluation of compressive strength of Euphorbia tortilis cactus infused M25 concrete by using ABAQUS under static load, *Mater. Lett.,* 2023, p. 135600.
https://doi.org/10.1016/j.matlet.2023.135600
27. Velumani, M., Mohanraj, R., Krishnasamy, R., and Yuvaraj, K., Durability evaluation of cactus-infused M25 grade concrete as a bio-admixture, *Period. Polytech. Civ. Eng.,* 2023, vol. 67, pp. 1066–1079.
https://doi.org/10.3311/PPci.22050

Publisher's Note. Pleiades Publishing remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.