

Fluorine Free Ph8 POSS Coated on Polyurethane Foam for Enhanced Oil/Water Separation Capability

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Abstract

The increasing use of fluorinated polyhedral oligomeric silsesquioxane (POSS) coupling agents in hydrophobic absorbent coatings has raised serious environmental concerns due to their persistence and toxicity. In this study, we report the synthesis of Fluorine-Free Octaphenyl POSS (F-FOPP) through hydrolysis and condensation reactions of phenyltrichlorosilane. The as-prepared nanohybrid was applied to polyurethane foam (PUF) using a facile, “one-pot” dip-coating method. ^1H -NMR, ^{13}C -NMR and ^{29}Si -NMR spectroscopic techniques confirmed the structure of the nanohybrid. F-FOPP demonstrated good thermal stability with pronounced decomposition at 450°C and char yields reaching 42%. Cytotoxicity evaluation revealed a non-toxic F-FOPP. Properties of the modified foam was analyzed using SEM, AFM, WCA measurement and UTM. The F-FOPP formulation at a concentration of 0.5 wt%, when applied to PUF imparted hydrophobicity with WCA exceeding 100° and enabled moderate oil/water separation in multiple cycles while maintaining good mechanical durability profile. This optimizable, fluorine-free approach enables the development of sustainable, absorbent coatings with hydrophobic and separation properties.

1 Introduction

Oil pollution is one of the global environmental challenges threatening ecosystems and human well-being as a result of environmental degradation caused by oil exploration, accidental spills, industrial operations, and the discharge of oily wastewater from petrochemical, textile, and food industries [1–3]. The presence of oil in aquatic environments can adversely affect water quality, aquatic organisms, and human health. Consequently, several clean-up strategies have been developed for the removal of oil from contaminated water. However, many conventional approaches are associated with high operational costs, complex processing requirements, and the potential generation of secondary pollutants [4,5]. Among the available remediation technologies, the use of highly porous oil absorbent materials has attracted considerable attention because of their simplicity, rapid response, ease of operation, and potential for oil recovery and reuse. In this regard, polymeric foams have been extensively investigated for oil/water separation and oil spill remediation because their interconnected porous structures provide abundant spaces for oil uptake and retention.

Superhydrophobic foams, characterized by a water contact angle greater than 150° , have received particular attention because their low surface energy and hierarchical surface structures can facilitate water repellency and selective oil uptake. Such foams have therefore been explored for applications in oil spill clean-up, oil/water separation, wastewater treatment, and related environmental remediation processes [6–11]. However, most superhydrophobic foams have been fabricated using fluorinated compounds and coatings. Although fluorinated materials can provide low surface energy, their widespread application has raised significant environmental persistence, bio-accumulation and toxicity concerns [12–14]. For example, per- and polyfluoroalkyl substances (PFAS) are highly persistent and resistant to environmental and biological degradation. Their persistence, mobility, and potential for bioaccumulation have generated considerable concern regarding their long-term environmental impact

and possible adverse effects on human health [15–17]. These limitations have therefore stimulated increasing research interest in the development of fluorine-free (F-F) coatings which may serve as alternative to hazardous fluorinated compounds.

A number of F-F superhydrophobic treatments have recently been reported [18]. For example, Mukherjee et al. developed a sustainable three-dimensional superhydrophobic and superoleophilic polyurethane foam coated with rice husk-derived silica and reduced graphene oxide, which exhibited a water contact angle of 164° , excellent mechanical and chemical stability and oil/water separation efficiencies exceeding 98% [19]. Most recently, Dharmaraj et al. developed a fluorine-free, highly durable Ph-POSS-ZIF-8 sponge and demonstrating its potential as an environmentally friendly alternative to fluorinated materials for oil/water separation applications [20]. In another work, Dharmaraj et al. developed eco-friendly porous composite of octaphenyl POSS and HKUST-1 with hydrophobic/oleophilic properties for oil and organic solvent sorption [21,22]. Till date, there has not been any report that take advantage of independently synthesized pure octaphenyl POSS directly coated on PU foam for oil/water separation, without graphene, metal organic framework, zeolite imidazolate framework or another secondary nanomaterial. Our research group has previously adopted POSS incorporated expanded polystyrene waste as nanofibrous membranes coated on hollow ceramics for both oil/water separation and eventual water purification [23]. The success of this approach is attributed to the incorporation of POSS, which enables effective modulation of surface properties, thereby enabling the oil/water separation performance.

In this study, we have hydrolytically condensed phenyltrichlorosilane to obtain F-FOPP in a facile chemistry approach. F-FOPP coated foam has hydrophobic surface capable of separating diesel oil-contaminated water in up to five absorption cycles. On the overall evaluation, F-FOPP demonstrated good thermal and mechanical stability and may serve as alternative to fluorinated silsesquioxane coupling agents.

2 Materials and methods

2.1 Materials

Pure and high grade phenyltrichlorosilane was procured from Sigma-Aldrich. KOH, toluene and all other commercially available reagents and solvents were used without further purification. Diesel oil was purchased from a local fuel station in Abeokuta. Pristine polyurethane foam was pre-cleaned by ultrasonication in ethanol for 30 min, followed by multiple rinses with distilled water, and subsequently dried in an oven at 80°C for further use.

2.2 Characterization

^1H , ^{13}C and ^{29}Si NMR spectroscopic data were obtained at ambient temperature using Bruker Avance III HD 300 spectrometer (operating at 500 MHz for ^1H , 125 MHz for ^{13}C and 59.63 MHz for ^{29}Si). According to the residual solvent resonance of CDCl_3 (1H, δ 7.24; ^{13}C , δ 77.0) C_6D_6 (1 H, δ 7.15), chemical shifts

are reported in values. The surface morphology of the synthesized silsesquioxane and modified PUF were observed by a SEM (Quanta 200 F, HV 15.0 kV). The microstructural property of the modified PUF was performed in bright-field mode using a TEM (JEOL JEM-2100, operating at 120 kV). The WCA of the modified PUF was measured at 3 different positions on the foam surface, where an OCA 15 Plus apparatus (DataPhysics Instruments, Filderstadt, Germany) outfitted with a high-resolution camera and an automatic liquid dispenser was used to measure the dynamic and static contact angles. Tensile property of the modified PUF was evaluated using an Instron 4486 UTM, with test conducted at ambient temperature and a crosshead speed of 5 mm min⁻¹. In addition, thermal stability test was conducted using a TGA (Mettler-Toledo with a heating rate of 10°C/min).

2.3 Synthesis of F-FOPP

The synthesis of octaphenyl POSS was carried out as shown in Scheme 1(i), where potassium hydroxide (0.3 g) was dispersed in 40 mL of toluene in a dried 100 mL round-bottom flask under a nitrogen atmosphere. Phenyltriethoxysilane (3.6 mL) was then added to the reaction mixture, which was refluxed at 105°C for 12 h. Subsequently, 0.5 mL of water was introduced at 3 h intervals, repeated three times [21]. The resulting white precipitate was collected by filtration, washed 7 times with deionized water and methanol, and dried at 80°C for 6 h to obtain F-FOPP (Yield = 79%). ¹H NMR (500 MHz, CDCl₃): δ (ppm) = 7.70–7.42 (m, 40H). ¹³C NMR (125 MHz, CDCl₃): δ (ppm) = 134 – 128. ²⁹Si NMR (59.63 MHz, CDCl₃): δ (ppm) = – 67.4.

2.4 Fabrication of F-FOPP Coated PUF

The fabrication process was carried as described by [24]. F-FOPP solution was prepared by adding F-FOPP (0.2 g) to ethanol (50 mL) and thoroughly mixed to ensure uniform dispersion. Cleaned PUF (2 x 2 x 2 cm) was immersed in the solution at 25°C with constant stirring for 30 min to achieve complete wetting. After immersion, the PUF was carefully removed with tweezers, squeezed, and then dried at 80°C for 1 h to obtain F-FOPP coated PUF as shown in Scheme 1(ii). The degree of surface functionalization was quantified by the weight gain ratio (WGR), calculated according to Eq. 2:

$$WGR = \frac{W_{after} - W_{before}}{W_{before}} \times 100\% \quad (Eq. 2)$$

where W_{after} and W_{before} represent the weight of the PUF after and before treatment, respectively. For PUF treated with 0.5 wt.% F-FOP, the WGR was determined to be 6.2%, indicating that the coating constituted approximately 6% of the modified PUF's total weight.

2.5 Thermal Stability of F-FOPP

Thermal stability testing was carried out by placing a small F-FOPP sample on a highly sensitive balance within a furnace, and its mass was continuously recorded as the temperature is ramped according to a programmed profile. Sample was heated from 30°C to 800°C at a constant heating rate of 10°C min⁻¹

under a nitrogen atmosphere with a purge flow of 50 mL min⁻¹ [25]. The percentage weight loss was calculated according to Eq. 1:

$$\text{Weight Loss (\%)} = \frac{W_o - W_t}{W_o} \times 100 \quad (Eq. 1)$$

where: W_o and W_t represent Initial weight and Weight at temperature (t), respectively.

2.6 In-Vitro Cytotoxicity Evaluation of F-FOPP

Cytotoxicity of F-FOPP was determined in-vitro by the incubation of mouse neuroblastoma (N2a) with or without 1.0 mM F-FOPP for 48 h using MTT assay [26]. The conversion of tetrazolium salt (MTT) to a coloured formazan by mitochondria and dehydrogenases is a marker of cell viability, which was determined. Detection of apoptotic and necrotic cells was also performed through propidium iodide staining. As cells can be determined by their DNA contents, cells were incubated with propidium iodide fluorescent dye that binds to DNA. This was followed by the analysis of the fluorescent intensity with Becton Dickinson LSR II flow cytometer. Subsequently, cell population histogram for N2a cell when treated with 1.0 mM F-FOPP and untreated (control) were determined.

2.7 Mechanical Stability of F-FOPP Coated PUF

The tensile strength testing of F-FOPP coated PUF was evaluated following standard procedure [27]. Before testing, the PUF was trimmed into rectangular strips (30 mm × 5 mm) and conditioned at room temperature for 10 h to minimize variability due to moisture or thermal effects. The conditioned PUF was then mounted in the upper and lower grips of the tensile tester and gauge length set to 20 mm. Tensile load was applied until the point of fracture and the resulting stress-strain data was recorded. Tensile stress (σ , MPa) was calculated according to Eq. 3:

$$\sigma = \frac{F}{A} \quad (Eq. 3)$$

where F and A represent Applied force (N) and Cross-sectional area (mm²), respectively. Strain (ϵ , %) was calculated according to Eq. 4:

$$\epsilon = \frac{L - L_o}{L_o} \times 100 \quad (Eq.4)$$

where: L_o and L represent Original gauge length and Extended length, respectively.

2.8 Oil Absorption Capacity of F-FOPP Coated PUF

Oil absorption capacity of F-FOPP coated PUF was determined following established sorption testing procedures [28]. Prior to testing, the foam was dried and weighed to obtain the initial dry mass. Thereafter, the PUF was immersed into an oil/water mixture (1:1 v/v) and swirled to ensure complete wetting. Upon reaching saturation, the PUF was removed and allowed to drain for 30 s. The oil-saturated PUF was weighed to obtain the wet mass. After weighing, the saturated foam was manually squeezed

with consistent pressure, weighed and reused for the next absorption cycle. The oil absorption capacity was calculated according to Eq. 5:

$$\text{Oil absorption capacity } (Q) = \frac{W_1 - W_o}{W_o} \qquad (Eq. 5)$$

where: W_o and W_1 represent mass before immersion and mass after oil saturation respectively.

4 Results and discussion

4.1 Structural Characterization of F-FOPP

The synthesized F-FOPP was characterized adequately using spectroscopic techniques such as ^1H -NMR, ^{13}C -NMR, ^{29}Si -NMR. The spectra found in Fig. 1 largely confirmed the successful synthesis and structural integrity. Apart from the ^1H -NMR and ^{13}C -NMR signals which strongly supported successful incorporation of phenyl ring into POSS framework, ^{29}Si -NMR spectra exhibited single peak signal at -67.4 ppm which is an indication that the nanohybrid not only have one structural conformation of silicon atom but also have uniform structure with high purity. Similar results have previously been reported [29–31]. Furthermore, these chemical shift values agree with the expected region for an aromatic-substituted cubic POSS at -65 to -70 ppm, supporting T_8 cage structural scaffold.

4.2 Evaluation of Thermal Stability of F-FOPP

TGA curve (Fig. 2) of F-FOPP demonstrated good thermal stability, with negligible weight loss observable below 200°C. However, significant thermal degradation commenced above 200°C, followed by pronounced decomposition between 250°C and 400°C, which can be attributed to the degradation of the organic moieties of the POSS structure. At temperatures above 500°C, the mass stabilized at 40%, suggesting the formation of a thermally stable siloxane (Si-O-Si) network as shown in Table 1. These results confirm that F-FOPP possesses adequate thermal stability for applications involving temperatures below 200°C [32].

Table 1
Thermal Stability Characteristics of F-FOPP from TGA

F-FOPP				
Temperature (°C)	200	400	600	800
Residual mass (%)	98	65	42	40

4.3 Cytotoxicity Profile of F-FOPP

The cell cycle analysis was carried out to evaluate the biocompatibility and cytotoxicity of F-FOPP. Figure 3 shows the cell cycle distribution of 2B cells after 48 h exposure to 1 mM F-FOPP. The DNA

content histogram of the treated cells almost completely overlaps with that of the untreated control. The values ($N = 0.7\%$; $V = 94.2\%$) as shown in Fig. 3, indicate that the vast majority of the cells remain viable, while only very small fraction undergoes cell death suggesting excellent cytocompatibility. Additionally, the negligible shift in DNA content distribution indicated sustainability due to maintaining of membrane integrity and normal cellular processes [33,34].

4.4 Surface Morphology of F-FOPP and F-FOPP coated foam

The surface morphology of F-FOPP and F-FOPP coated PUF were characterized by SEM, revealing a significant structural differences. The F-FOPP micrograph shows an irregular, rough and aggregate particulate morphology as shown in Fig. 4A. The absence of a smooth, uniform surface suggests that F-FOPP exists as nanostructures material with a high surface area. This morphology is beneficial for surface modification because the rough particles can provide additional micro/nanoscale features when coated unto the PUF surface. Likewise, the surface of the PUF coated with F-FOPP became noticeably rougher, as shown in Fig. 4B. The SEM image of the coated PUF shows that the original open-cell structure of the PUF is retained after coating [35]. However, the struts of the PUF appear rougher and more textured than would be expected for uncoated PUF, indicating deposition of F-FOPP onto the foam skeleton. Furthermore, the coating appears to introduce additional surface irregularities. Such roughness can enhance water repellency of a hydrophobic surface by reducing the effective contact area between water and the solid surface [36]. The AFM image further confirms that the coated PUF possesses a non-uniform, textured surface at the micro/nanoscale as shown in Fig. 4C. The observable variations in surface topography indicate that deposition of F-FOPP alters the surface architecture of the PUF. The resulting rough surface can contribute to enhanced hydrophobicity because surface roughness amplifies the intrinsic wetting characteristic of the coating. Figure 4D shows the WCA measurement of F-FOPP coated PUF, the modified foam exhibits water contact angle of 105.3° . Since the contact angle is greater than 90° , the modified PUF can be classified as hydrophobic. This suggests a clear change from a comparatively more water-wettable surface and confirms that F-FOPP successfully alters the surface wettability of PUF [37].

4.5 Evaluation of Mechanical Stability of F-FOPP

Although hydrophobic surfaces have been widely investigated, the poor mechanical stability is a major setback to their further development. Therefore, it is necessary to evaluate the stability of coated foam sample to ensure their durability in oil/water separation applications. The stress-strain curve of F-FOPP coated PUF (Fig. 5) shows the mechanical response of the foam under tensile loading. The stress-strain curve demonstrates that the modified foam possesses appreciable mechanical strength and deformation capability. The tensile stress increased progressively with strain, reaching a maximum value of 11MPa at 20% strain. The gradual increase in stress reflects progressive deformation and stretching of the polymeric foam network. Beyond the maximum tensile stress, a sharp decrease in stress was

observed at 22% strain, indicating structural failure of the foam as shown in Table 2. The combination of tensile strength and high strain tolerance suggests that the F-FOPP coating preserves the mechanical integrity of the polyurethane substrate while providing a modified surface suitable for oil/water separation [38].

Table 2
Mechanical Stability Characteristics of F-FOPP from UTM.

F-FOPP				
Strain (%)	5	10	15	20
R Tensile stress (MPa)	3.4	6.4	9.0	11.0

4.6 Oil Absorption Capacity of F-FOPP Coated Foam

To evaluate the potential of the coated foam in diesel oil/water separation applications, we utilized a gravimetric analysis. The oil absorption capacities of F-FOPP modified foam over 5 cycles are presented in Fig. 6. The modified foam exhibited an initial diesel-oil absorption capacity of 4.8 g/g in the first cycle. The capacity increased slightly to 5.8 g/g in the second cycle, which may be associated with progressive wetting of the porous foam and better penetration of diesel into the interconnected pores. However, after the second cycle, a gradual decline was observed. The absorption capacity decreased to 3.6 g/g in the third cycle, 2.9 g/g in the fourth cycle and 1.8 g/g in the fifth cycle. The reduction may result from incomplete removal of the previously absorbed oil during squeezing, partial pores blockage and/or some loss or redistribution of the F-FOPP coating during repeated mechanical manipulation. On the overall, the material demonstrated reusability but with noticeable loss of absorption capacity over repeated cycles [39,40].

5 Conclusion

This study successfully developed a fluorine-free octaphenyl polyhedral oligomeric silsesquioxane as a sustainable alternative to fluorinated silsesquioxane for the fabrication of hydrophobic polyurethane foam through a facile, “one-pot” dip-coating process. Multinuclear NMR (¹H, ¹³C, and ²⁹Si) confirmed the successful synthesis of the silsesquioxane coupling agent while preserving the integrity of the silsesquioxane T₈ cage, whereas SEM and AFM analyses verified the formation of uniform micro/nanostructured coating with increased surface roughness. The resulting water contact angle of F-FOPP coated foam (105.3°) confirmed the transformation of the foam surface into a hydrophobic state, which is favourable for selective interaction with oil during oil/water separation. The thermal analysis indicated that the material maintained good thermal stability at temperatures below 200°C, while significant decomposition occurred at higher temperatures, with substantial silicon-containing residue remaining at elevated temperatures. The mechanical analysis further showed that the F-FOPP coated foam could withstand considerable tensile deformation, reaching a maximum tensile stress of 11 MPa at 20% strain, demonstrating the surface functionalization did not eliminate the mechanical integrity of

the porous PU substrate. The oil absorption studies demonstrated the ability of the F-FOPP coated foam to absorb diesel oil. Although, the absorption capacity decreased to 3.6, 2.9 and 1.8 g/g in the third, fourth and fifth cycles, respectively, the foam retained measurable oil absorption capacity after repeated regeneration. More importantly, F-FOPP exhibited excellent biological compatibility, maintaining a cell viability of 94.2 with only 0.7 necrotic cells. These results establish that F-FOPP coated PU foam is a promising porous material for oil/water mixture separation.

Declarations

Declarations

Ethics approval

We wish to clarify that no human participants or animals were directly involved in this study. The cytotoxicity evaluation was performed using an established Neuro-2a (N2a), mouse neuroblastoma cell line obtained from the American Type Culture Collection (ATCC) and maintained under the recommended culture conditions. Therefore, ethical approval and informed consent were not required for this study.

Consent to participate

This is not applicable. This study did not involve human participants and therefore no informed consent was required. All experiments were performed on the synthesized F-FOPP and F-FOPP coated PUF in the laboratory.

Consent for publication

This is not applicable. The manuscript does not include any data from human participants or animals. All results are derived from laboratory-based experiments on the F-FOPP and F-FOPP coated PUF and no personal or sensitive information is involved.

Competing interests

The authors declare that they have no conflicts of interest, financial or personal, that could have influenced the research, analysis, or interpretation of the results reported in this manuscript.

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Author Contribution

IOD: Conceptualization, Investigation, Methodology, Visualization, Writing–original draft. DAB: Investigation, Visualization, Writing–original draft. AAB: Investigation, Visualization, Writing–original draft. FA: Investigation, Methodology, Visualization. JAA: Investigation, Methodology, Visualization. SOA: Methodology, Validation, Writing–review & editing. FOO: Methodology, Validation, Writing–review & editing. ORA: Investigation, Validation, Visualization. EOD: Conceptualization, Methodology, Writing–review & editing, Supervision.

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Data Availability

All data generated and analyzed during this study are included within this article. No additional datasets were generated or are required to support the conclusions of this work.

References

1. M. Sonbhadra, L.M. Pandey, Bioelectrochemical remediation and valorization of oily wastewaters: A review, *J. Hazard. Toxic Radioact. Waste* 29 (1) (2025) 03124001, <https://doi.org/10.1061/JHTRBP.HZENG-1421>
2. L.A. Mokif, H.K. Jasim, N.A. Abdulhusain, Petroleum and oily wastewater treatment methods: A mini-review, *Mater. Today Proc.* 49 (2022) 2671–2674, <https://doi.org/10.1016/j.matpr.2021.08.340>
3. T. Alomar, B.H. Hameed, M.A. Al-Ghouti, F.A. Almomani, D.S. Han, A review on recent developments and future prospects in the treatment of oily petroleum refinery wastewater by adsorption, *J. Water Process Eng.* 64 (2024) 105616, <https://doi.org/10.1016/j.jwpe.2024.105616>
4. Y. Zhang, X. Li, H. Wang, J. Chen, Recent advances in superwetting materials for efficient oil/water separation: Fabrication strategies and environmental applications, *Chem. Eng. J.* 462 (2023) 142289, <https://doi.org/10.1016/j.ccej.2023.142289>
5. M.S.M. Mansour, H.I. Abdel-Shafy, A.M. Ibrahim, Petroleum wastewater: Environmental protection, treatment, and safe reuse: An overview, *J. Environ. Manage.* 351 (2024) 119827, <https://doi.org/10.1016/j.jenvman.2023.119827>
6. H. Lee, G. Chen, B.P. Chang, T.H. Mekonnen, Recent progress in the development of porous polymeric materials for oil ad/absorption application, *RSC Appl. Polym.* 3 (1) (2025) 43–77, <https://doi.org/10.1039/D4LP00211C>
7. S.M. Hailan, I. Krupa, G. McKay, Removal of oil spills from aqueous systems by polymer sorbents, *Int. J. Environ. Sci. Technol.* 22 (2025) 3833–3854, <https://doi.org/10.1007/s13762-024-05959-0>

8. 8. P. Pi, Z. Ren, Y. Yang, W. Chen, Y. Lin, A review of various dimensional superwetting materials for oil–water separation, *Nanoscale* 16 (2024) 17248–17275, <https://doi.org/10.1039/D4NR01473A>
9. 9. J. Qi, S. Li, X. Guo, Z. Fang, Y. Liu, Advancements in superhydrophobic foam materials for efficient oil-water separation: Modification techniques and future applications, *J. Water Process. Eng.* 70 (2025) 107008, <https://doi.org/10.1016/j.jwpe.2025.107008>
10. 10. T. Li, Y. Peng, H. You, X. Guan, J. Lv, C. Yang, Recent developments in the fabrication and application of superhydrophobic surfaces, *Chem. Rec.* 24 (9) (2024) e202400065, <https://doi.org/10.1002/tcr.202400065>
11. 11. Z. Wang, H. Ma, B. Chu, B.S. Hsiao. Super-hydrophobic polyurethane sponges for oil absorption. *Separation Science and Technology.* 22 (2017); 52(2):221-7, <https://doi.org/10.1080/01496395.2016.1246570>
12. 12. I.T. Cousins, J. DeWitt, J. Glüge, Z. Wang, M. Scheringer, The high persistence of PFAS is sufficient for their management as a chemical class, *Environ. Sci. Process. Impacts* 25 (2023) 1094–1110, <https://doi.org/10.1039/D3EM00129K>
13. 13. X. Yin, Y. Zhang, H. Li, L. Wang, J. Chen, Fluorine-free superhydrophobic materials: Recent advances, challenges and future perspectives, *Chem. Eng. J.* 489 (2024) 151345, <https://doi.org/10.1016/j.cej.2024.151345>
14. 14. P. Loganathan, R. Yogapriya, A. Chinnusamy, K.K. Datta, S. Shanmugan. In situ growth of octa-phenyl polyhedral oligomeric silsesquioxane nanocages over fluorinated graphene nanosheets: super-wetting coatings for oil and organic sorption. *Dalton Transactions.* (2025) 13;54(3):1150-63, <https://doi.org/10.1039/D4DT02678K>
15. 15. S. Alhassan, M. Numan, M. Al-Ghouti, Current state of knowledge of environmental occurrence, toxic effects, and advanced treatment of PFOS and PFOA, *Sci. Total Environ.* 913 (2024) 169332, <https://doi.org/10.1016/j.scitotenv.2023.169332>
16. 16. E.J. Itumoh, S. Data, J.L.-Y. Chen, M. Kah, L.P. Padhye, E.M. Leita, Addressing the persistence of per- and poly-fluoroalkyl substances (PFAS): Current challenges and potential solutions, *RSC Sustainability* 2 (2024) 3183–3201, <https://doi.org/10.1039/D4SU00152D>
17. 17. I. Rosato, T. Bonato, T. Fletcher, E. Batzella, C. Canova, Estimation of per- and polyfluoroalkyl substances (PFAS) half-lives in human studies: A systematic review and meta-analysis, *Environ. Res.* 242 (2024) 117743, <https://doi.org/10.1016/j.envres.2023.117743>
18. 18. H. Zhang, L. Li, M. Wang, C. Guo, F. Liu, Y. Zang, C. Chen, Fluorine-free fabrication of durable superhydrophobic membranes for efficient oil–water separation, *Sci. Rep.* 16 (2026), <https://doi.org/10.1038/s41598-026-48607-y>
19. 19. M. Mukherjee, S.K. Ghadei, B. Rakesh, U. Balaji, R. Sakthivel, K.J. Sankaran, Fluorine-free sustainable three-dimensional superhydrophobic and superoleophilic robust foam for efficient oil/water separation, *ACS Appl. Eng. Mater.* 2 (2) (2024) 324–334, <https://doi.org/10.1021/acsaenm.3c00626>

20. K. Dharmaraj, M. Sanjay Kumar, S. Sathish, M. Prakash, S. Shanmugan. Octaphenyl Polyhedral Oligomeric Silsesquioxane-Driven In Situ Growth of Corrosive-Resistant and Hydrophobic–Oleophilic ZIF-8 Nanostructures for Oil–Water Separation. *ACS Applied Nano Materials*. (2025) 14;8(6):2897 – 911, <https://doi.org/10.1021/acsanm.4c06571>
21. K. Dharmaraj, M. Sanjay Kumar, N. Palanisami, M. Prakash, P. Loganathan, S. Shanmugan. Eco-friendly porous composite of octaphenyl polyhedral oligomeric silsesquioxane and HKUST-1 with hydrophobic–oleophilic properties towards sorption of oils and organic solvents. *Dalton Transactions*. (2024) 13;53 (32):13602-16. <https://doi.org/10.1039/D4DT01397B>
22. H. Zhang, Y. Liu, X. Wang, J. Li, L. Chen, J. Zhao, Fluorine-free bio-inspired superhydrophobic coating based on stearic acid for durable self-cleaning and oil/water separation applications, *Sci. Rep.* 15 (2025) 92246, <https://doi.org/10.1038/s41598-025-92246-8>
23. F.O. Oladoyinbo, D. Elebiju, F. Akinwunmi, O. Ejeromedoghene, Y.A. Alli, S.O. Alayande, S.A. Amolegbe, A.R. Olayide, D. Díaz Díaz, E.O. Dare, Ceramic bowl-supported nanofibrous membrane with fluorinated silsesquioxane-inspired switchable surfaces for successive crude oil/water separation and secondary water treatment, *Colloids Surf. A Physicochem. Eng. Asp.* 686 (2024) 133224, <https://doi.org/10.1016/j.colsurfa.2024.133224>
24. A.L. Ruschel, W. Pro, M.R. Begley, F.W. Zok, Simulating effects of microstructure on the mechanical response of flexible foams, *J. Mater. Res.* 40 (2025) 2323–2336, <https://doi.org/10.1557/s43578-025-01658-9>
25. E.H. Verbon, J.A. Post, J. Boonstra, The influence of reactive oxygen species on cell cycle progression in mammalian cells, *Gene* 511 (1) (2012) 1–6, <https://doi.org/10.1016/j.gene.2012.09.040>
26. Y. Wang, R. Pan, J. Zhang, J. Qian, J.-M. Basset, F. Jérôme, G. Mignani, D. Sun, L. Zhou, Fluorine-free POSS-based silane coatings for robust superhydrophobic and self-cleaning cotton fabrics with efficient oil–water separation performance, *J. Appl. Polym. Sci.* 143 (2026) e58085, <https://doi.org/10.1002/app.58085>
27. D. Walicki, P. Zawistowski, J. Ryszkowska, Exploring the microstructure–property relationship in polymer foams using advanced statistical methods, machine learning and deep learning: A review, *Comput. Mater. Sci.* 256 (2025) 113909, <https://doi.org/10.1016/j.commatsci.2025.113909>
28. Y. Ma, M. Maihaiti, M. Mamat, S. Feng, Oil–water separation via superhydrophilic materials: Fundamentals and perspectives for 2D/3D system design, *J. Environ. Chem. Eng.* 13 (2025) 119564, <https://doi.org/10.1016/j.jece.2025.119564>
29. M. Laird, C. Carcel, M. Wong Chi Man, J.R. Bartlett, A ring-strain model for predicting ^{29}Si NMR chemical shifts in polyhedral oligomeric silsesquioxanes and siloxanes, *Dalton Trans.* 54 (2025) 4856–4860, <https://doi.org/10.1039/D4DT03515A>
30. M. Laird, P. Gaveau, G. Silly, C. Carcel, M. Unno, J.R. Bartlett, M. Wong Chi Man, Functionalised polyhedral oligomeric silsesquioxane with encapsulated fluoride—First observation of fluxional $\text{Si}\cdots\text{F}$ interactions in POSS, *Dalton Trans.* 49 (2020) 6236–6244, <https://doi.org/10.1039/C9DT04725A>

31. 31. M. Chen, Y. Wang, M. Wang, W. Zhou, POSS-based hybrid biomaterials for tissue engineering and regenerative medicine, *Mater. Today Bio* 32 (2025) 101837, <https://doi.org/10.1016/j.mtbio.2025.101837>
32. 32. G.L. Song, Z. Feng. Modification, degradation and evaluation of a few organic coatings for some marine applications. *Corrosion and Materials Degradation*. 2020 Dec 21;1(3), <https://doi.org/10.3390/cmd1030019>
33. 33. A. Kwiatkowski, T. Rucinski, A. Jabłońska, A. Michałowicz, Per- and polyfluoroalkyl substances (PFAS)—Occurrence, toxicity and biodegradation in the environment: A review, *Chemosphere* 347 (2024) 140627, <https://doi.org/10.1016/j.chemosphere.2023.140627>
34. 34. Y. Liu, L. Shi, H. Wang, X. Zhao, Toxicological effects and mechanisms of per- and polyfluoroalkyl substances on human health: A comprehensive review, *J. Hazard. Mater.* 475 (2024) 135006, <https://doi.org/10.1016/j.jhazmat.2024.135006>
35. 35. Y. Chen, H. Wang, X. Zhang, L. Wen, P. Pi, X. Wen, Rational design and application of superhydrophobic fluorine-free coating based on double-decker silsesquioxane for oil-water separation, *Polymer* 304 (2024) 127143, <https://doi.org/10.1016/j.polymer.2024.127143>
36. 36. M. Prakash, S. Sathish, S. Shanmugan, Octaphenyl polyhedral oligomeric silsesquioxane-driven *in situ* growth of corrosive-resistant and hydrophobic–oleophilic ZIF-8 nanostructures for oil–water separation, *ACS Appl. Nano Mater.* 8 (2025) 2897–2911, <https://doi.org/10.1021/acsanm.4c06571>
37. 37. X. Hu, Y. Zhang, H. Chen, Y. Li, Fluorine-free, self-healing, and superhydrophobic coating for efficient oil/water separation and fog-water collection, *Polym. Adv. Technol.* 35 (2024), <https://doi.org/10.1002/pat.6584>
38. 38. J. Peng, Y. Zhang, Modification of octavinyl POSS and its effect on the mechanical properties and thermal stability of silicone rubber/POSS composites, *Nanomaterials* 15 (22) (2025) 1706, <https://doi.org/10.3390/nano15221706>
39. 39. K.M. Lichade, A.A. Deshpande, Y. Pan, 3D Printing of Hierarchical Porous Structures for Oil/Water Separation and Absorption, *ACS Appl. Polym. Mater.* 7 (17) (2025) 11425–11434, <https://doi.org/10.1021/acsapm.5c01215>
40. 40. J. Zhao, M. Li, W. Liao, Y. Shao, Q. Du, D. Li, Y. Nan, Z. Zhang, D. Sun, Hierarchically porous degradable foam prepared by water-in-oil high internal phase emulsion template for high-performance recyclable oil–water separation, *J. Hazard. Mater.* 500 (2025) 140385, <https://doi.org/10.1016/j.jhazmat.2025.140385>

Scheme 1

Scheme 1 is available in the Supplementary Files section.

Figures

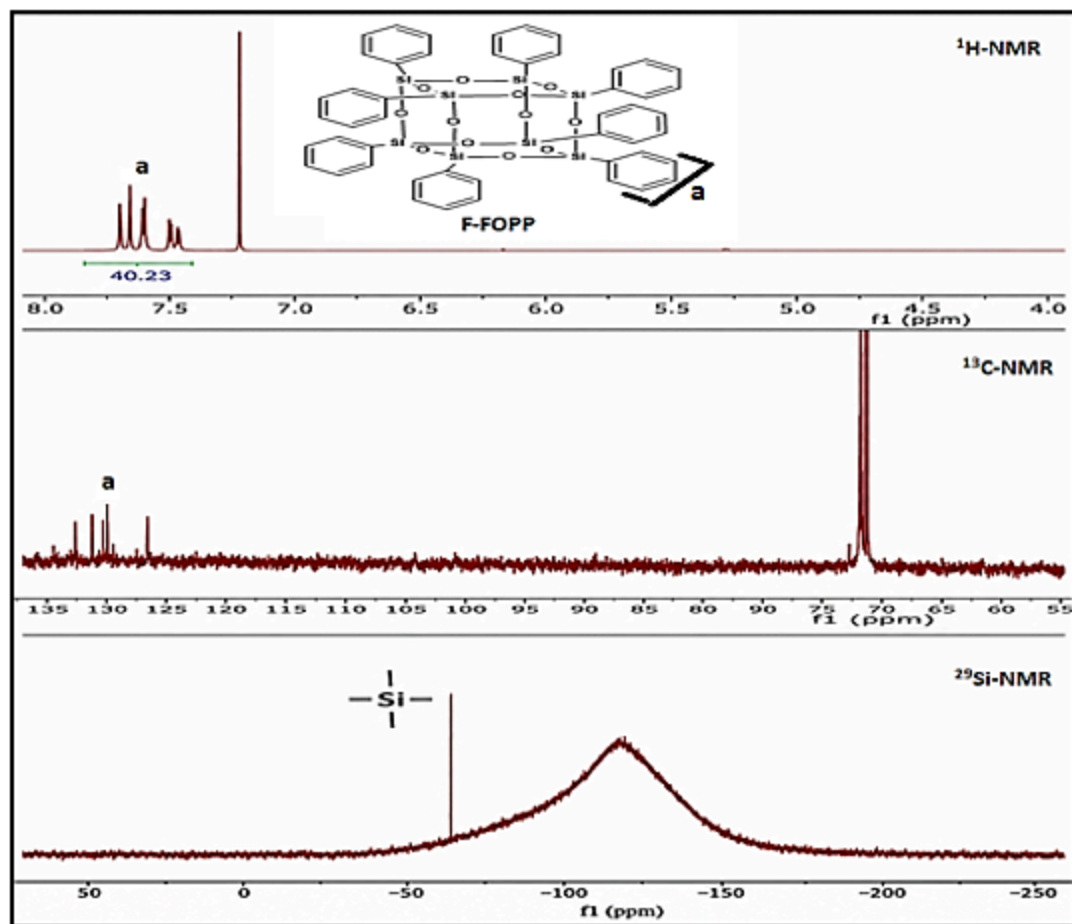


Figure 1

^1H NMR, ^{13}C NMR and ^{29}Si -NMR Spectra of F-FOPP.

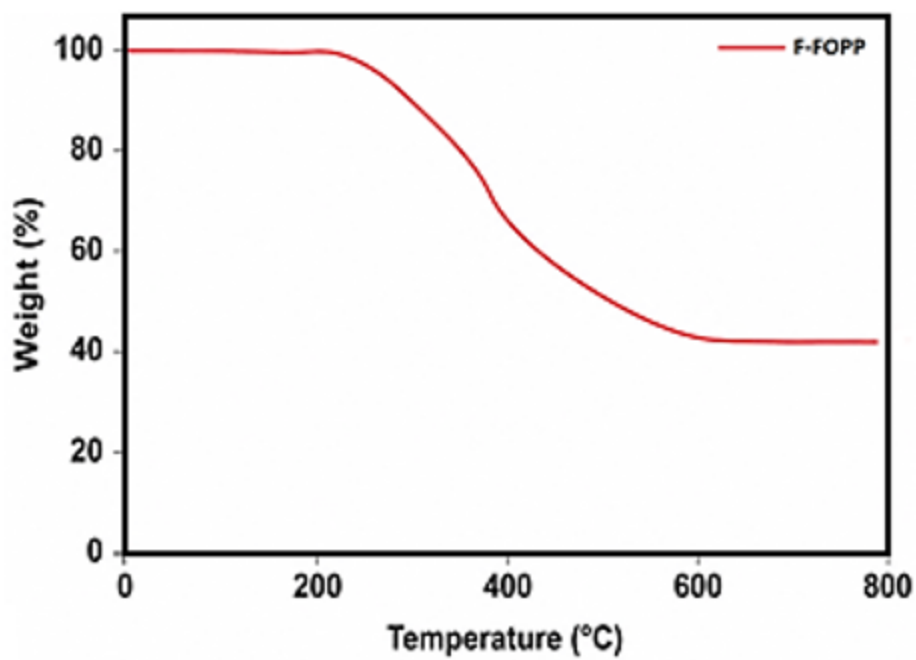


Figure 2

Thermal Stability Curve of F-FOPP.

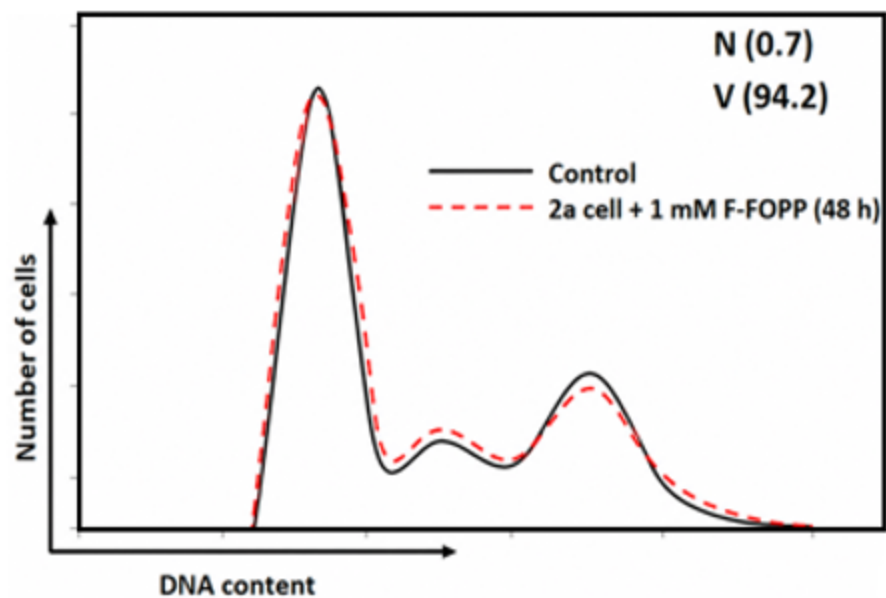


Figure 3

Cell Cycle Distribution and DNA Content of F-FOPP Cell Treatment.

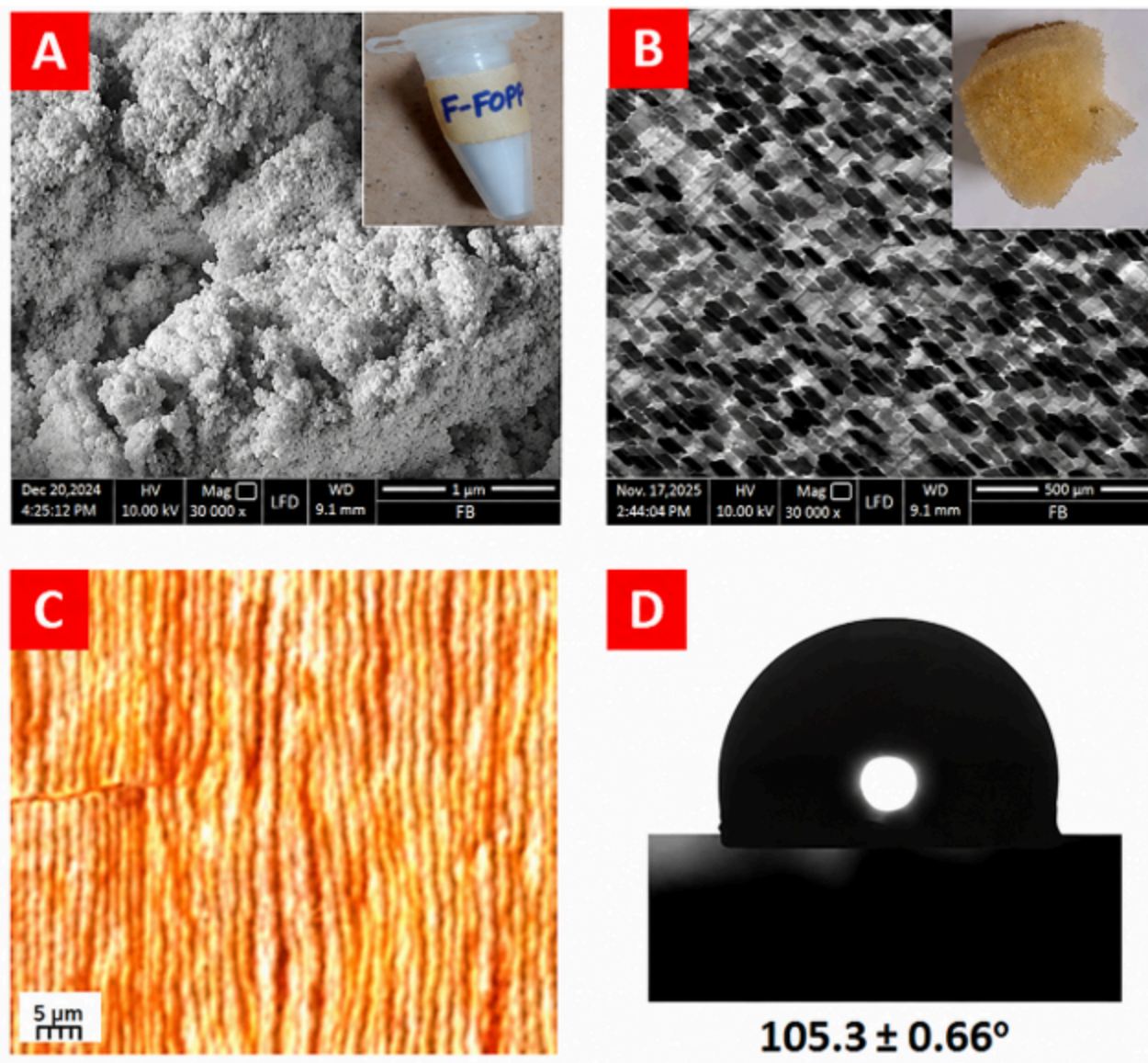


Figure 4

(A) SEM Micrograph of F-FOPP (B) SEM Micrograph of F-FOPP Coated PUF (C) AFM Micrograph of F-FOPP Coated PUF and (D) WCA Measurement of F-FOPP Coated PUF.

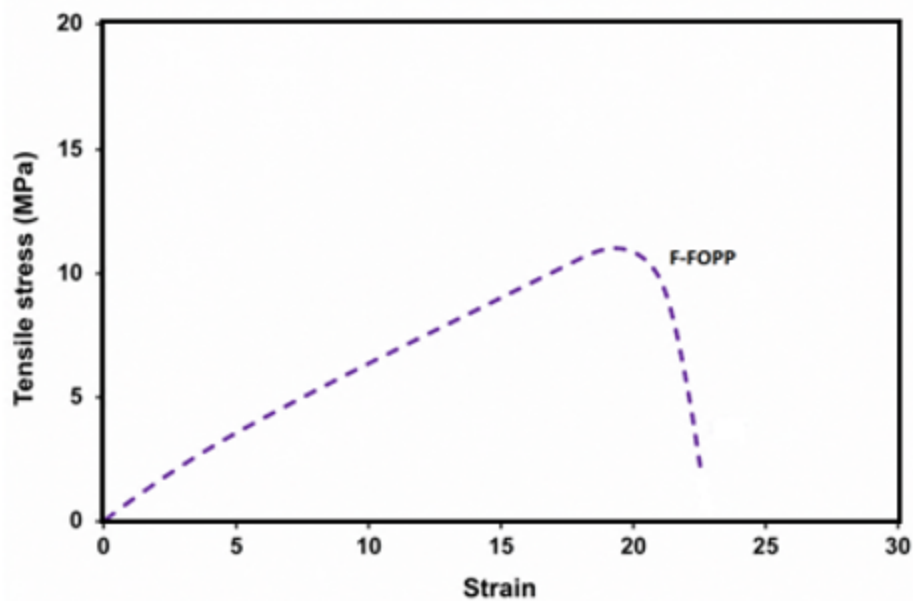


Figure 5

Stress-Strain Curve of F-FOPP Coated Foam.

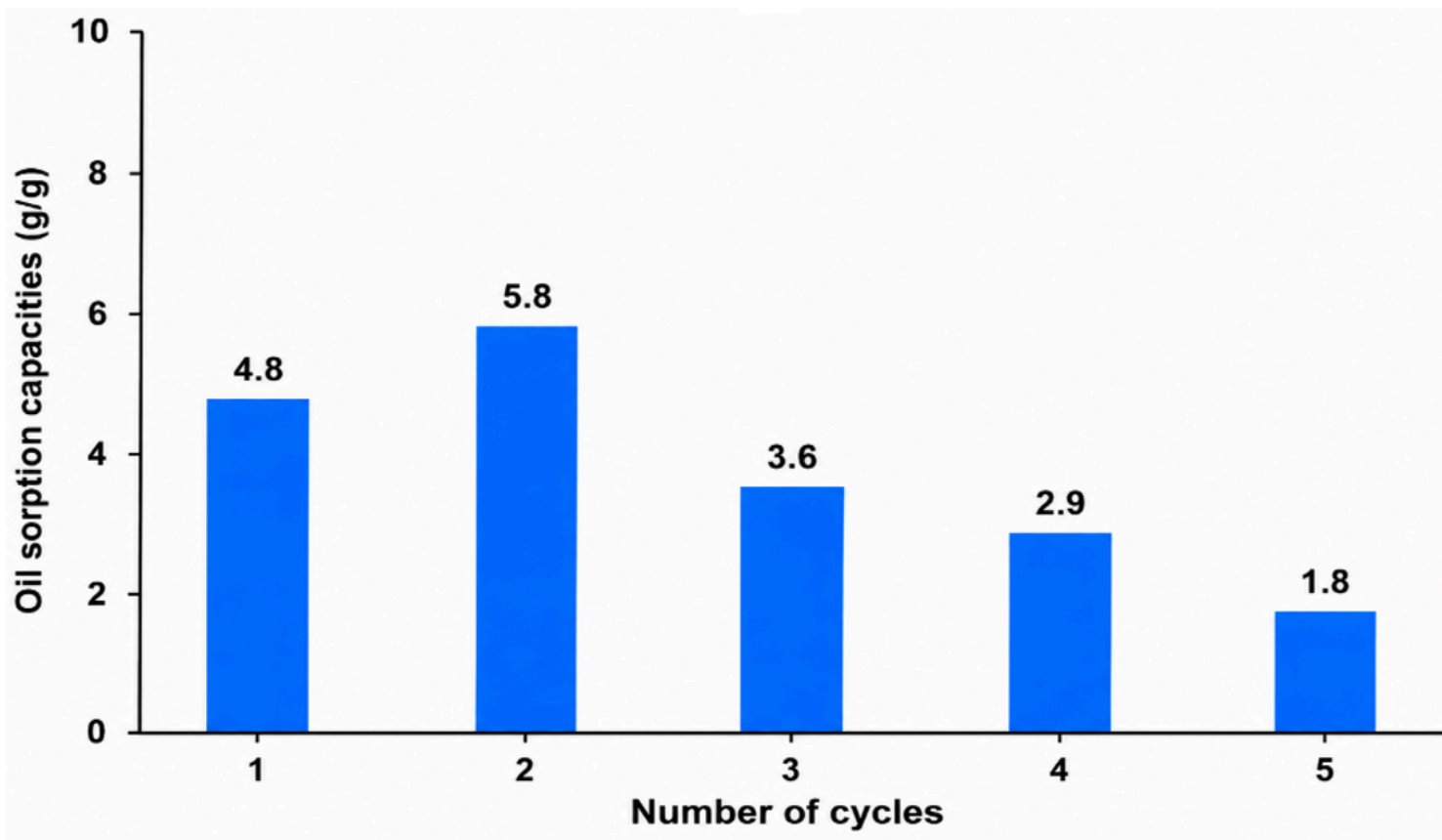


Figure 6

Diesel Oil Absorption Capacity of F-FOPP Coated Foam.

Supplementary Files

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- [Scheme1.png](#)