Journal of Basics and Applied Sciences Research (JOBASR)
ISSN (print): 3026-9091, ISSN (online): 1597-9962
Volume 4(3) June 2026 Special Issue
DOI: https://dx.doi.org/10.4314/jobasr.v4i3.14s

Iron-trimesate Impregnated Moss Composites for Application in Petroleum Recovery and Remediation of Petroleum-contaminated Water

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ABSTRACT

Crude oil spills are major environmental and economic concerns. The use of sorbents for removal of crude oil from water surfaces has remained a viable solution to crude oil spills. In this study, a novel composite material produced by impregnation of the metal-organic framework (MOF), iron-trimesate (Fe-tri), onto the moss species, *Barbula indica*, was investigated for its crude oil sorption capacity. Various moss/Fe-tri composition, including moss/Fe-tri 50:50, moss/Fe-tri 75:25, and moss/Fe-tri 25:75, were prepared and tested along with the pure substances for their capability to sorb crude oil. Infrared spectrometric analyses carried out for moss/Fe-tri 50:50, pure moss sample, and pristine unmodified Fe-tri (MOF) revealed characteristics bonds and functional groups such as -OH, -C=O, C-H and aromatic rings on the materials. The results obtained from the crude oil sorption tests showed relatively high sorption capacity values, which decreased in the order: moss/Fe-tri 75:25 (2.81 g/g) > pure moss sample (2.39 g/g) > moss/Fe-tri 50:50 (2.32 g/g) > moss/Fe-tri 25:75 (2.09 g/g) > Fe-tri (1.68 g/g), indicating that the impregnation of moss with Fe-tri enhanced crude oil retention capacity. Water sorption test results further demonstrated the suitability and efficiency of the materials as sorbents for removal of crude oil from water and also indicated that sorbent porosity and pore volume influenced sorption performance. The findings revealed that impregnating mosses with metal-organic frameworks could yield a composite with better crude oil retention property, and highly efficient for application in recovery of crude oil from water and remediation of water contaminated with same.

Keywords:
Sorption,
Crude oil,
Moss,
Iron-trimesate

INTRODUCTION

Petroleum has inspired arguably the most convenient technologies to utilize energy for mankind's socio-economic benefits (Eisler, 2024). Thus, the demand for the substance has gone astronomically high, and its production has turned into the industry of focus in many nations (Adewuyi, 2016).

Petroleum spills are inevitable occurrences in petroleum production (Wekpe et al., 2024). The major consequences of these spills have remained the associated environmental damages and economic losses amongst others (Li et al., 2016; Ordinioha & Brisibe, 2013). Post petroleum spill activities are all efforts by petroleum producing companies towards petroleum recovery and cleanup of spill sites (Ukhurebor et al., 2023). Oil usually disperse in water forming emulsions and creating petroleum product recovery difficulties (Mueller et al., 2025). Membrane based sorption technologies have been identified as the most reliable methods for coping with the challenges of oil/water separations (Awwad et al., 2023). Oil spill response technologies operated by petroleum production companies rely heavily on the use of sorbents and solidifiers to recover petroleum from spill sites (*TIP8UseofSorbentMaterialsinOilSpillResponse.Pdf*, n.d.). The US National Oil and Hazardous Substance Pollution Contingency Plan (NCP), under Subpart J, section 300.915, define sorbents as inert and insoluble materials used to remove oil and hazardous materials from water through adsorption,

How to cite this article: Ukachuku S. & Igiegie O. D. (2026). Iron-trimesate Impregnated Moss Composites for Application in Petroleum Recovery and Remediation of Petroleum-contaminated Water. *Journal of Basics and Applied Sciences Research*, *4*(3), 130-139. https://dx.doi.org/10.4314/jobasr.v4i3.14s

in which the oil or hazardous substance is attracted to the sorbent surface then adheres to its surface or absorption, in which the oil or hazardous substance penetrates the pores of the sorbent material, or a combination of the two phenomena (*40 CFR 300.915 -- Data and Information Requirements for Listing on the NCP Product Schedule or Sorbent Product List.*, n.d.). Some of the conventional sorbents for oil spill treatment include: natural sorbents such as peat moss, corn cobs, cellulose fibers, and animal-based such as bird feathers; inorganic based sorbents such as, zeolite and volcanic ash; and synthetic sorbents such as, polypropylene, polyethylene, polyurethane, and polyester ("Application of Sorbents and Solidifiers for Oil Spills," 2007). Sorbents may be applied directly to contaminated areas as loose materials or powders, in woven or felted mats, or deployed as booms where the sorbent fillings are encased in long lengths of mesh material (Núñez-Delgado et al., 2021).

The merits of the conventional natural and synthetic sorbents are reportedly cost effectiveness, eco-friendliness, and abundance. However, the problem of poor performance efficiency in petroleum uptake and recovery continue to emerge, due mainly to poor surface area, poor porosity, high hydrophilicity, and low hydrophobicity (Shahmirzaee et al., 2022). This has led to an increased search for better alternatives (Zamparas et al., 2020), which is also the goal of this study.

In this study, we are targeting the mechanical combination of two known ultra-porous materials, moss and metal-organic framework, MOF. The rationale is to carry out a mechanical blending of a little quantity of the MOF with a large quantity of the moss. Various moss/MOF combination ratio will be investigated for their oil sorption performance. While moss is a non-vascular plant of the division Bryophyta known for its high fluid absorption and retention capacity (Pandey & Alam, 2019), MOFs are hybrid materials consisting of metal nodes that are linked by organic polytopic ligands, forming a network of interconnected, multi-dimensional micro-cages that can function as hosts to chemical forms (Elhenawy et al., 2020). Moss reportedly exhibit appreciably high buoyancy suitable for application as sorbents for petroleum on water surfaces (Pandey & Alam, 2019). The representative moss in this study is a native of the study area, known as *Barbula indica* (Wall Screw Moss). *Barbula indica* is commonly found in the study local on concrete surfaces, especially of abandoned and unmaintained buildings, and thrives in moist/humid conditions (Etim & Folarin, 2009).

The proposed impregnation of MOF into moss is intended for enhancement of porosity, surface area and hydrophobicity. The anticipated advantageous properties of the proposed moss/Fe-MOF composite include buoyancy (a property required of sorbents, so they can remain on the water surface together with the crude oil, for the sorption to take place), mechanical strength (required for adsorbent stability in both water and in crude oil), ultrahigh surface area, high pore volume (characteristics of MOFs (Elhenawy et al., 2020)), as well as increased fluid retention property.

Iron (Fe) based MOF was chosen for this study upon consideration of the toxicity characteristics of Fe as well as its role in facilitating bioremediation of surface waters contaminated with petroleum. Iron has been reported to exhibit an extremely low to negligible toxicity profile, since it is a micronutrient required for various biochemical and physiological functions (Tchounwou et al., 2012), thus it is an essential element to organisms. Further, if Fe (III) leaches into a water body, it most likely forms insoluble oxides and hydroxides that precipitates (Cardwell et al., 2023), making it even less available for ingestion by organisms. In remediation of petroleum contaminated water, iron acts as a bioavailable electron acceptor for anaerobic biodegradation, and also an adsorbent for oil removal (Castro et al., 2022). Iron-based remediation techniques, particularly utilizing iron nanoparticles (FeNPs) or iron oxides, enhance the degradation of total petroleum hydrocarbons (TPH), including polycyclic aromatic hydrocarbons (PAHs) Muthukumar et al., 2024; Castro et al., 2022).

MATERIALS AND METHODS

The reagents and analytical instruments for the study were iron nitrate ($Fe\,(NO_3)_2 \cdot 6H_2O$), a product of Aladdin Biological Technology Co, LTD, Shanghai, China; trimesic acid (1,3,5-benzenetricarboxylic acid), a product of Merck KGaA, Darmstadt, Germany, N,N-dimethyl formamide and methanol, both sourced from Merck KGaA, Darmstadt, Germany, rounded bottom flask, refluxing apparatus, electrically powered blender, a porcelain mortar/pestle, and scan infrared spectrometer (Buck M500 Quick-Scan IR spectrometer).

Moss plant, *Barbula indica*, was plucked from a concrete made fence around the study area and was carefully filtered to separate impurities, thoroughly washed and rinsed with sterile water, then sun dried. The dry moss biomass was then ground to fine particulate form using an electrically powered blender.

Crude oil sample was collected from an oil field located in Otuokpoti, Ogbia Local Government Area, Bayelsa State, Nigeria. Then, stored in a sealed container at room temperature. Nylon mesh nets of mesh size 3 micron were used as model booms to wrap the sorbents in the crude oil sorption studies. 500 mL beakers were employed as crude oil spill simulation tanks, while the measuring cylinders were used to transfer predetermined volumes of water and crude oil. A high precision analytical balance from PJ electronics, model BPJ-6001, Seoul, South Korea was used for all weight measurements in the study. Sterile water was obtained from the distillery in the Chemistry laboratory, University of Africa, Toru-Orua, for use in the preparation of crude oil spill simulation tanks.

Preparation of Iron-trimesate framework (Fe-tri)

Iron-trimesate framework was prepared according to the method described by Dikio and Farah, (2013), with some modifications (Dikio & Farah, 2013). 14.4 g of Iron nitrate hexahydrate and 10.5 g of trimesic acid were weighed and introduced into a round bottom flask containing 50 mL of N,N-dimethyl formamide (DMF). The mixture was refluxed at a temperature of 140 ^{0}C for 12 hours. The product was then washed with methanol, centrifuged, oven dried at minimal temperature, and transferred to a storage container.

Preparation of Fe-tri-impregnated moss

The Fe-tri-impregnated moss materials (moss/Fe-tri) were prepared via a mechanical mixing of the pure ground moss and the as-synthesized Fe-tri framework using a porcelain mortar and a pestle. 5 g each of three distinct types of moss/Fe-tri materials were prepared, including moss/Fe-tri (25:75), moss/Fe-tri (50:50), and moss/Fe-tri (75:25), with moss/MOF weight ratio of 1.25 g/3.75 g, 2.5 g/2.5 g, and 3.75 g/1.25 g respectively. The photographs are presented in Figure 1.

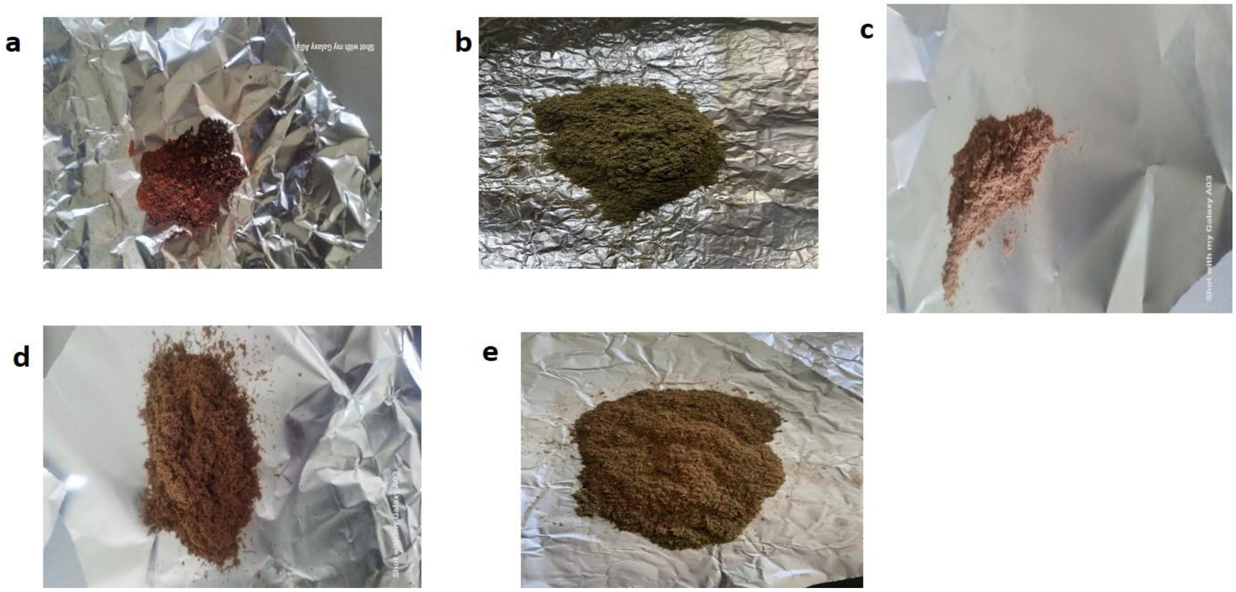

Figure 1. Photograph of the investigated materials. a. pristine unmodified Fe-tri. b. pure moss. c. moss/Fe-tri (50:50). d. moss/Fe-tri (25:75. e. moss/Fe-tri (75:25)

Infrared characterization

Infrared characterization was carried out for moss/Fe--tri (50:50), pure moss, and pristine unmodified Fe-tri, separately. 1g material was weighed into an agate mortar. Few drops of Nujol (mineral oil) were added and mixed with the aid of the agate pestle. Resulting mixture was dispersed on and sandwiched between NaCl cells and recorded in scan infrared spectrophotometer between 4000 cm^{-1} and 600 cm^{-1}.

Preparation of crude oil spill simulation tanks

Five sets of crude oil spill simulation tanks were prepared using 500 mL beakers. The tanks were prepared by adding 20 ml (~ 18 g) of crude oil (density ~ 0.9gcm^{-1}) into 200 ml of distilled water. Initial weight of oil/water mixture in each tank was recorded

Petroleum sorption studies

Petroleum sorption tests were carried out according to the method prescribed in ASTM F726-17 (Standard Test Method for Sorbent Performance of Adsorbents for use on Crude Oil and Related Spills) (F20 Committee, 2017). 1.2 g of each sorbent type was weighed in triplicate and wrapped in nylon mesh nets of mesh size 3 micron. Crude oil sorption tests were carried out in three cycles for each sorbent, consecutively, with fresh sorbent wrap used in every cycle, in the same simulation thank and in the same state it was left after a prior cycle. Each sorbent wrap was dipped into crude oil spill simulation tank for a direct contact between floating crude oil and contents of the wrap. Contact was maintained for 10 minutes and upon removal from the tank, unsorbed oil was allowed to drain off (into the tank) for 1 minute. After every cycle, weight of oil/water content of simulation tank was recorded.

Weight of sorbed oil was estimated by weight differences, as follows:

Weight (in grams) of sorbed oil = (Initial weight (grams) of oil/water mixture) – (Final weight (grams) oil/water mixture). Equation 1

Water sorption studies

In the water sorption test, 1.2 g of each sorbent type was wrapped in a mesh net and dipped into a beaker containing a known weight of distilled water and allowed to remain for 10 minutes. Upon, lifting up the soaked wrap, draining of unabsorbed water was allowed for 1 minute. The weight of water absorbed by each sorbent was estimated by weight difference between initial and final weight of water inside the beaker.

RESULTS AND DISCUSSION

Infrared Characterization results

Figure 2a shows the IR spectra of pure moss sample. It has been reported that bryophytes consist of hemicellulose and pectin (30–60%), cellulose (15–25%), proteins (5–10%), polyphenols (5–10%) and lastly inorganic substances (3–10%) (Orlov & Sadovnikova, 2005). The broad band ranging from 3,564 to 3024 cm^{-1} represents vibrations of –OH groups from cellulose like compounds in the moss. The peak seen after the broad band at 2937 to 2873 cm^{-1} represents C−H stretching vibrations. Further, the band at 1760 to 1619 cm^{-1} is typical of carbonyl function (-C= O). These carbonyl functions in mosses are from ketones, esters, carbohydrates and proteins present in the biomass. The band between 1529 and 1312 cm^{-1} could be due to -OH bending vibrations, vibrations of aromatic skeletons as well as aromatic C−H vibrations. While the band at 1260 to 928 cm^{-1} are from C−C and C−O from cell wall polysaccharides (Kļaviņa, 2018a).

Figure 2b shows the infrared spectra of pristine unmodified Fe-tri (MOF). The broad band observed between 3500 and 3,000 cm^{-1} represent -OH stretching vibration of water molecules. The peaks observed at 1700 to 1400 cm$^{−1}$ is related to the carboxylate anion which attaches Fe (II) ion. More precisely, the bands spanning 1452 to 1273 cm$^{−1}$ correspond to the asymmetric and symmetric stretching vibrations of the carboxylate groups in trimesic acid (García et al., 2014). Further, stretching vibration and bending vibration of C-H bonds of benzene rings are seen at 2925 to 2835 cm^{-1} and 1184 to 940 cm$^{−1}$ respectively. While the narrow peak at 672 cm^{-1} corresponds to vibrations of Fe-O bonds.

Figure 2c represents IR spectra of moss/Fe-tri 50:50. The broad band at 3,541 to 3,001 cm^{-1} represents O–H stretching vibrations. Those at 1760 to 1542 cm^{-1}, 1414 to 1260 cm^{-1}, 1222 to 940 cm^{-1} and 697 to 620 cm^{-1} depicts the carbonyl/carboxylate functions, aromatic rings, C–H groups, and Fe–O vibrations respectively.

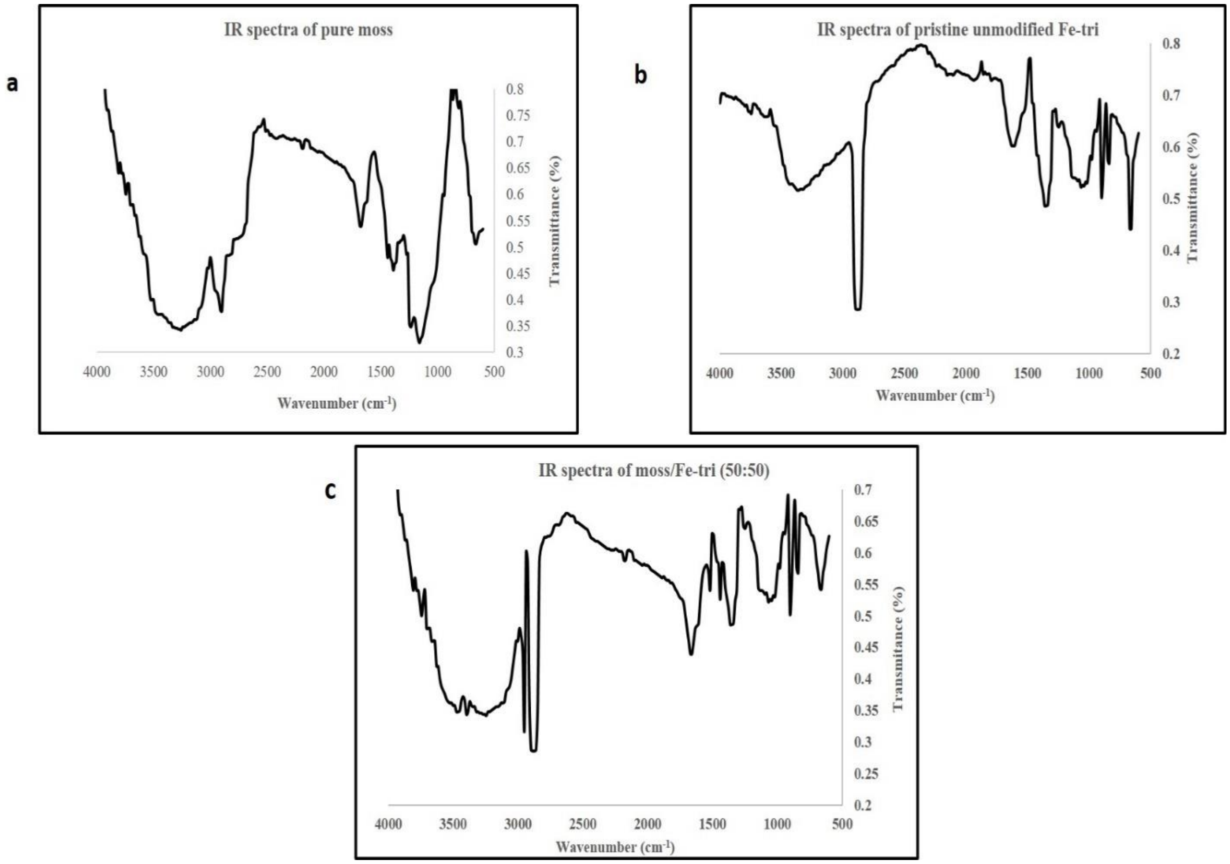

Figure 2. IR spectra of sorbents. a. pure moss. b. pristine unmodified Fe-tri. c. moss/Fe-tri (50:50)

Petroleum and water sorption tests

The results of the petroleum sorption test are presented in terms of the crude oil sorption capacity of each sorbent used. The crude oil sorption capacity was estimated with the equation of Nwadiogbu and his co-workers (2016), as shown in Equation 2 below (Nwadiogbu et al., 2016).

$$\text{Oil sorption capacity} = \frac{\text{net weight of oil sorbed}}{\text{weight of dry sorbent}} \quad \text{Equation 2.}$$

The percentage crude oil sorbed after three cycles of sorption tests was calculated as:

$$\%\ \text{crude oil sorbed} = \frac{\text{net weight of sorbed oil}}{\text{initial weight of crude oil during first cycle of test}} \times 100\% \quad \text{Equation 3.}$$

The results of crude oil sorption test obtained from the three cycles of tests carried out are presented in Table 1-3. The net sorbed weight of crude oil, net percentage of sorbed crude oil, and the mean sorption capacity of each sorbent are presented in Table 4 and Figure 3

The results of the water sorption tests for each of the sorbents are also presented in Figure 4

Table 1. Crude oil sorption capacity and percentage sorption for the various sorbent types (cycle 1)

S/N	Sorbent type	Initial weight of crude oil (g)	Final weight of crude oil (g)	Weight of sorbed crude oil (g)	Crude oil sorption capacity of sorbent (g/g)	% crude oil sorbed	
1	Moss/Fe-tri 50:50	18	14.55	3.45	2.88	19.17	
2	Moss/Fe-tri 25:75	18	15.36	2.64	2.20	14.67	
3	Moss/Fe-tri 75:25	18	13.57	4.43	3.69	24.61	
4	Moss	18	14.64	3.36	2.80	18.67	
5	Fe-tri	18	15.70	2.30	1.92	12.78	

Table 2. Crude oil sorption capacity and percentage sorption for the various sorbent types (cycle 2)

S/N	Sorbent type	Initial weight of crude oil (g)	Final weight of crude oil (g)	Weight of sorbed crude oil (g)	Crude oil sorption capacity of sorbent (g/g)	% crude oil sorbed	
1	Moss/Fe-tri 50:50	14.55	11.66	2.89	2.41	19.86	
2	Moss/Fe-tri 25:75	15.36	12.75	2.61	2.18	16.99	
3	Moss/Fe-tri 75:25	13.57	10.03	3.54	2.95	26.09	
4	Moss	14.64	11.46	3.18	2.65	21.72	
5	Fe-tri	15.70	13.73	1.97	1.64	12.55	

Table 3. Crude oil sorption capacity and percentage sorption for the various sorbent types (cycle 3)

S/N	Sorbent type	Initial weight of crude oil (g)	Final weight of crude oil (g)	Weight of sorbed crude oil (g)	Crude oil sorption capacity of sorbent (g/g)	% crude oil sorbed	
1	Moss/Fe-tri 50:50	11.66	09.65	2.01	1.68	17.24	
2	Moss/Fe-tri 25:75	12.75	10.37	2.28	1.90	17.88	
3	Moss/Fe-tri 75:25	10.03	7.89	2.14	1.78	21..34	
4	Moss	11.46	09.40	2.06	1.72	17.98	
5	Fe-tri	13.73	11.94	1.79	1.49	13.04	

Table 4. Sorbent performance recorded for entire crude oil sorption test operation

S/N	Sorbent type	Initial weight of crude oil (g)	Net weight of sorbed crude oil (g)	mean crude oil sorption capacity of sorbent (g/g)	Net % crude oil sorbed
1	Moss/Fe-tri 50:50	18	8.35	2.32	46.39
2	Moss/Fe-tri 25:75	18	7.53	2.09	41.83
3	Moss/Fe-tri 75:25	18	10.11	2.81	56..17
4	Moss	18	8.60	2.39	47.78
5	Fe-tri	18	6.06	1.68	33.67

The mean crude oil sorption capacity of each sorbent is the mean weight of crude oil in grams sorbed per gram of sorbent, after three cycles of sorption tests. The values of the mean crude oil sorption capacity of each of the sorbents were 2.32, 2.09, 2.81, 2.39, and 1.68 g/g for moss/Fe-tri 50:50, moss/Fe-tri 25:75, moss/Fe-tri 75:25, pure moss and pristine unmodified Fe-tri (MOF) respectively. The net percentage of crude oil sorption after three cycles of sorption were: 46.39, 41.83, 56.17, 47.78, and 33.67% for moss/Fe-tri 50:50, moss/Fe-tri 25:75, moss/Fe-tri 75:25, pure moss and pristine unmodified Fe-tri (MOF) respectively. These results are represented in bar charts in Figures 3 and 4. As can be observed from the bar charts, the crude oil sorption capacity of sorbents in descending order is moss/Fe-tri 75:25 > pure moss > moss/Fe-tri 50:50 > moss/Fe-tri 25:75 > pristine unmodified Fe-tri. The observed trend in the sorption capacity of the sorbents underscores the effectiveness of mosses as fluid absorbent. The results clearly show that the larger contribution to crude oil sorption was provided by moss. Hence, among the three Fe-tri impregnated mosses, crude oil sorption capacity and percentage sorption increased with increasing moss weight. The higher crude oil sorption capacity of moss/MOF 75:25 relative to that of pure moss shows that the impregnation of moss with Fe-tri led to enhancement in crude oil retention property. This better crude oil retention property may be attributed to the hydrophobic sites on the MOFs used to impregnate the moss as well as the higher surface area of the composite relative the moss. MOFs exhibit ultra-high surface area as one of their intrinsic properties. Factors that reportedly enhance crude oil sorption capacity of a sorbent include any one, or a combination of, porosity, surface area of sorbent, and surface hydrophobicity/oleophilicity of the sorbent (Wang et al., 2023). Pristine unmodified Fe-tri (MOF) had the lowest crude oil sorption capacity (1.68 g/g) among the investigated sorbents. This could be indicative of a slower crude oil sorption rate, such that the material could not take up as much crude oil within the investigated time as compared to the other sorbents.

The results of the water sorption tests conducted for the various sorbents are presented in bar charts in Figure 5. The bar charts represent the water sorption capacity of each sorbent. The significance of the water sorption test is to evaluate the water sorption capacity of a potential petroleum sorbent, intended for application in water. This is because a low water sorption is essential for high sorbent performance in crude oil sorption, selectivity of crude oil over water, and indication of sufficient buoyancy of the material (Olga et al., 2014). The water sorption capacities of the sorbents in descending order were: moss/Fe-tri 75:25 (5.86 g/g) > pure moss (4.95) > moss/Fe-tri 25:75 (4.67 g/g) > moss/Fe-tri 50:50 (3.23 g/g) > pristine unmodified Fe-tri (1.95 g/g). The high water sorption of the moss-based sorbents highlights the dominant role of porosity and large pore volume in the uptake of the fluids. Influence of surface hydrophobicity/hydrophilicity was lesser. However, a combination of porosity and hydrophobicity was instrumental in the relatively high sorption capacity of moss/MOF 75:25 Mosses are reportedly high water absorbents with a high water retention capacity due to their high porosity and their hydrophilic surfaces (Kļaviņa, 2018b). The lower viscosity of water over crude oil contributed to the higher water sorption capacity of the sorbents relative to that crude oil. Pristine unmodified Fe-tri, with a water sorption capacity of 1.95 g/g exhibited a poorer water sorption/retention relative to the moss-based sorbents. This could be attributed to the dominant water repelling effect of the hydrophobic sites on the MOF, which are the benzene rings of the organic linkers. Overall, the crude oil sorption capacity of the investigated materials could present them as promising materials as sorbents for petroleum removal from water.

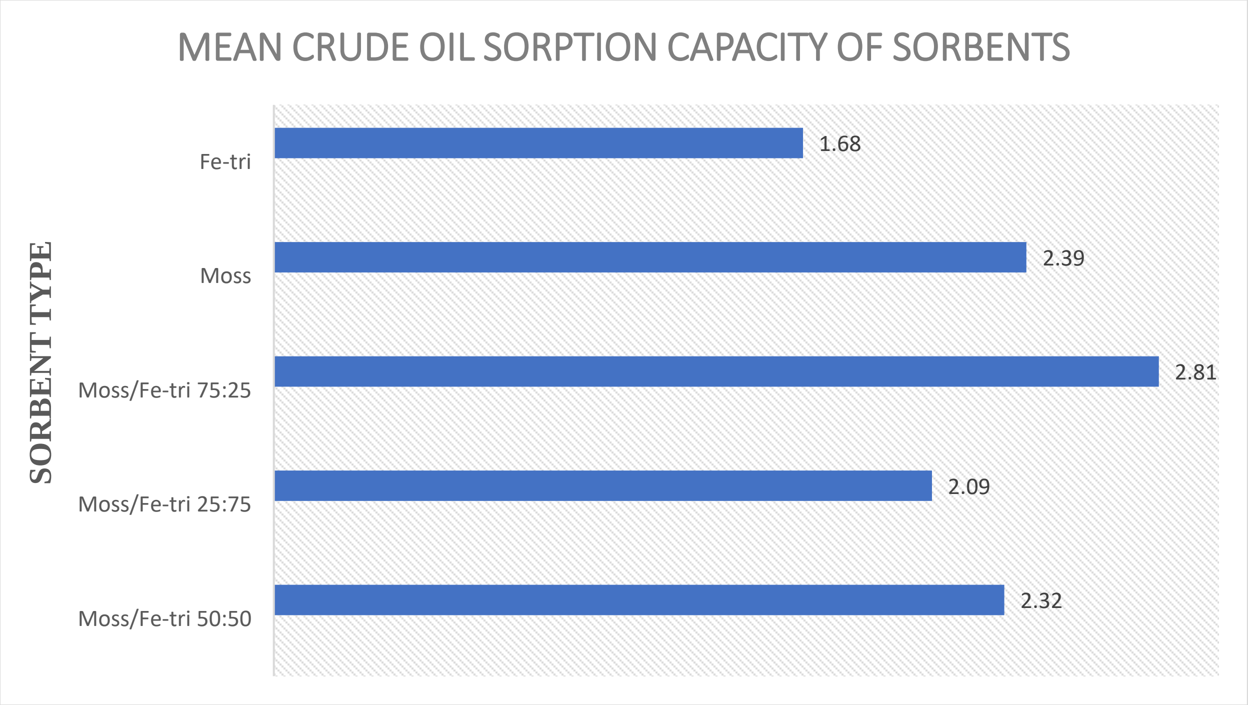

Figure 3. Bar chart representation of mean crude oil sorption capacity of sorbents (g/g)

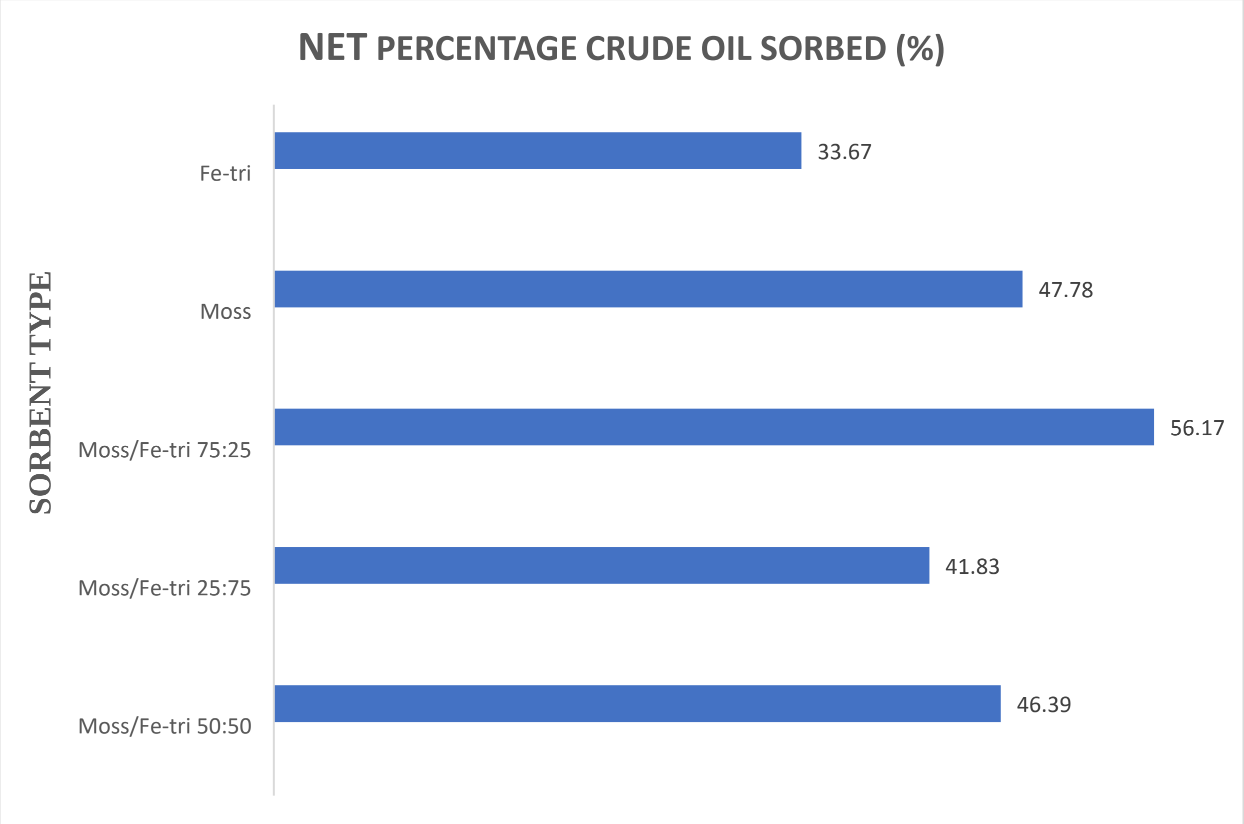

Figure 4. Bar chart representation of percentage crude oil sorption

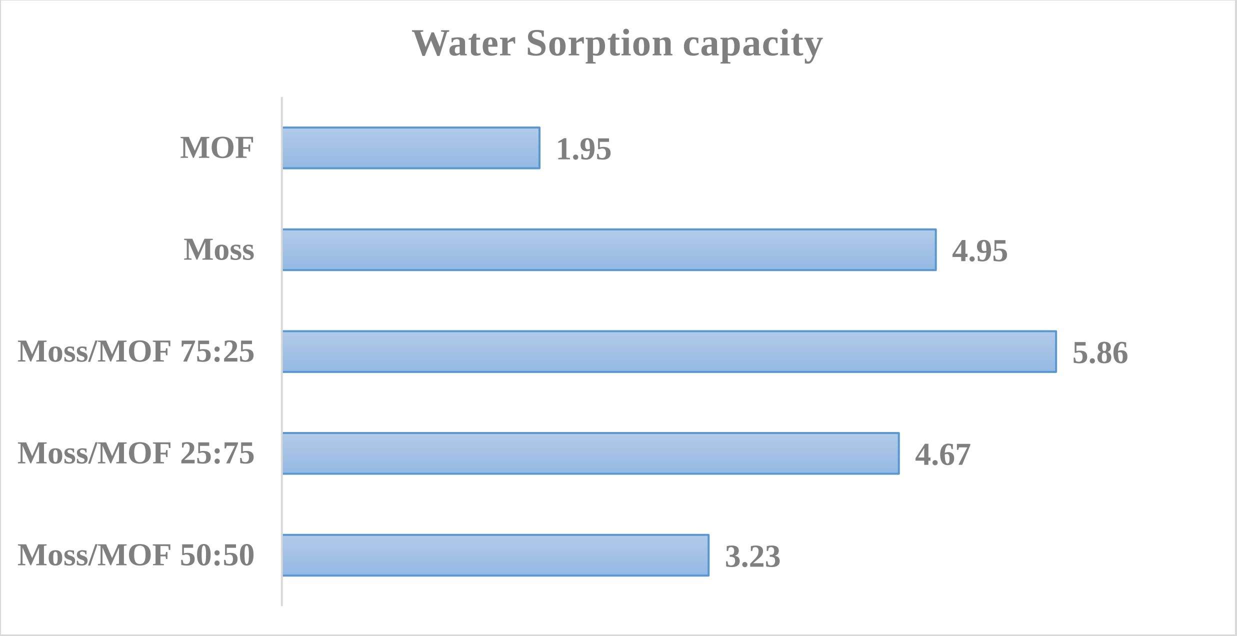

Figure 5. Bar chart representation of water sorption capacity of sorbents (g/g)

CONCLUSION

In this study, samples of the moss species *Barbula indica*, were impregnated with Iron-trimesate (Fe-tri), an iron based metal-organic framework (MOF). The resulting composites were tested for their crude oil sorption capacity. Various compositions of the composites were prepared including moss/Fe-tri 50:50, moss/Fe-tri 25:75, and moss/Fe-tri 75:25 and tested along with the pure moss and the pristine unmodified Fe-tri (MOF). One of the Fe-tri-impregnated mosses, moss/Fe-tri 50:50, along with pure moss sample, and the pristine unmodified Fe-tri (MOF) were subjected to infrared spectrometric characterization, to ascertain their characteristic functional groups. While the IR spectra for pure moss indicated the presence of hydroxyl (–OH), C−H, carbonyl (-C= O), C−C and C−O groups, that of moss/Fe-tri 50:50 showed the presence of hydroxyl (O–H), carbonyl/carboxylate anionic groups, aromatic rings, C–H groups, and Fe–O vibrations, and the pristine unmodified Fe-tri (MOF) showed carboxylates anionic groups, aromatic rings, C–H, and Fe-O bonds, which are indicative of the successful preparation of the metal-organic framework,.

The crude oil sorption tests were carried out in three cycles per sorbent, by dipping 1 g of fresh sorbent (in every cycle) wrapped in 3 micron mesh net, into crude oil spill simulation tanks containing 20 mL (~18 g) of crude oil in 200 mL of sterile water, and allowing an equilibration time of 10 minutes and a drain time of 1 minute.

Water sorption tests were also carried out for each of the sorbents to determine their suitability for application in water, and their efficiency as sorbents for removal of crude oil from water. Weight of sorbed fluid were recorded as weight difference between initial and final weights of beaker and the fluid contents.

The results from both crude oil sorption tests and water sorption tests showed that moss-based sorbents have excellent fluid sorption capacity owing to their large pore openings, pore volumes and surface functions. Impregnation of moss with Fe-tri (MOF) enhanced crude oil sorption and retention, with moss/Fe-tri 75:25 having the most crude oil sorption capacity 2.81 g/g, attributed to the combined effects of porosity and hydrophobicity. Pristine unmodified Fe-tri showed the lowest crude oil sorption capacity, 1.68 g/g due to a slow sorption of crude oil. However, it shows promises as an efficient material for crude oil retention if given sufficient sorption time.

We deduce from the results that the investigated materials could be established as sorbents for enhanced removal of petroleum and its products from water. However, recommends the following areas for further research: variation of sorbent/crude oil contact times, variation of sorbent dosages, variation of crude oil volume/weights in simulation tanks, and sorbent optimization studies utilizing established physicochemical methods for sorbent modification for improvement of their surface hydrophobicity.

Acknowledgments:

The authors wish to acknowledge the following persons: The Dean, Faculty of Basic and Applied Sciences, University of Africa, Toru-Orua, Professor Sikoki Francis, and the Chemistry Laboratory Technologists, of the same University Mr Luis Brave and Mr Daniel Ziriki who granted us access to the Laboratory space and Instruments to conduct this research.

Declaration of conflicts of Interest:
There is no conflict of interest to declare in this study.

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