RESEARCH ARTICLE

Self-Assembled Organic Aerogel and Sponges for Rapid and Effective Absorption of Oil from Oil-Contaminated Soil Samples

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ABSTRACT: Crude oil spills pose significant risks to both marine ecosystems and human populations. Traditional remediation techniques have their limitations, prompting the exploration of innovative approaches. This study introduces a novel method involving the synthesis of a self-assembled sponge and organic aerogel. These materials were further modified using hexamethyldisilazane (HMDS) and dodecyltrimethoxysilane (DTMS) to impart hydrophobic properties. Characterization via SEM examination and FTIR spectroscopy revealed distinctive features, including bands at approximately 1050 cm^{-1} and 790 cm^{-1}, indicative of alkyl side chain stretching by Si-O-C and C-H, respectively, enhancing the hydrophobicity of the materials. The SEM images depicted a random rough surface with abundant pores, facilitating oil adsorption from contaminated samples. Notably, after seven cycles of use with contaminated soil samples, the organic aerogel exhibited exceptional oil removal capacity, achieving 100% efficiency according to the Sears method. This innovative sponge and organic aerogel-based approach represents a promising advancement in environmental remediation, offering a highly efficient and sustainable solution for oil spill cleanup.

Keywords: Density functional theory; Photo-physical properties; Non-linear optical properties

1. INTRODUCTION

The search for effective and ecologically friendly ways to stop oil spills and soil contamination has taken center stage in an era marked by increased concerns about environmental sustainability and the need for creative solutions to manage pollution and contamination [1-3].

Oil contamination is a major worldwide environmental concern because to the rising need for oil and its derivatives as well as the inherent risks associated with its storage, transportation, and industrial activities [4-5]. Hydrocarbon spills that happen by accident or persistent seepage into soil ecosystems have serious consequences for biodiversity, soil fertility, and human health that call for immediate remedial actions. Traditional cleanup techniques can entail expensive

and time-consuming procedures like excavation and disposal, which have a significant negative impact on the environment and the economy [6, 7].

The objective of this study is to overcome the shortcomings of traditional remediation techniques by utilizing a novel combination of two exciting materials: organic aerogels that have self-assembled and specially made sponges. A powerful combination is produced when organic aerogels, which are renowned for their exceptional properties like large surface area, little density, and good oil absorption capacity are combined with sponges that have been especially created for this use [8–10]. These materials are excellent choices for effectively recovering oil from contaminated soil samples because of their exceptional affinity for hydrophobic chemicals.

Furthermore, the study emphasizes efficacy and speed heavily, offering a quick and very effective oil removal procedure that may drastically cut down on the time and expense of cleanup operations. This strategy reduces the overall environmental impact of cleanup activities, providing

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a workable option that not only meets the urgent requirement to recover contaminated soil but also aligns with sustainability goals [11, 12].

This study investigates the synthesis, characterization, and application of these materials as we go deeper into the domains of organic aerogels and creative sponge designs, providing a thorough grasp of the science underlying their remarkable performance [13, 14]. In order to examine the absorption kinetics, capacity, and selectivity of these materials in practical settings, the research makes use of state-of-the-art technologies and methodologies [15]. This will enable the creation of sustainable ways to counteract oil contamination in soil samples [16].

We shall delve into the complex realms of oil removal dynamics, sponge engineering, and organic aerogels in the pages that follow, highlighting their potential to transform environmental remediation. Through investigating the distinct characteristics and uses of these materials, this study aims to enhance our understanding and generate novel, environmentally-friendly methods for tackling the pressing problem of remediating oil-contaminated soil. The research summarized in the title has the potential to change how we approach the problems caused by oil spills and soil contamination on a worldwide scale, with implications for a cleaner, healthier, and more sustainable future.

## 2. EXPERIMENTAL DETAILS

2.1. Materials

From Sigma-Aldrich, all of the analytical-quality compounds were obtained. The following chemicals were used without being purified: triton X-100, formaldehyde, acetic acid, dodecyltrimethoxysilane (DTMS), acetonitrile, polyvinyl alcohol (PVA) powder, tetraethyl orthosilicate (TEOS, 99%), chitosan, acetic acid, ammonium hydroxide ($NH_3.H_2O$, 25.0%), sodium chloride, sodium hydroxide, and acetonitrile. The diesel fuel was obtained from the nearby gas station located in Hisar, India.

2.2. Synthesis of Hydrophobic Polyvinyl-alcohol formaldehyde sponge

In a 250 mL round bottom flask, 5.0 g of PVA powder and 45.0 mL of deionized water were thoroughly mixed to create the polyvinyl alcohol formaldehyde sponge. Using an oil bath, the entire solution was aggressively agitated for eight hours at 95 °C using a magnetic stirrer.   After that, the mixture was put into a 500 mL beaker and swirled once more while it was at room temperature for 15 minutes. The aforesaid PVA solution was then dropwise supplemented with 0.46 mL triton X-100 surfactant, 5.0 mL formaldehyde, and 1.5 mL concentrated sulphuric acid. Finally, the mixture was forcefully agitated to achieve the maximum foam. The achieved foam was transferred onto a glass petri dish and allowed to air dry for sixteen hours at 70˚C. To get rid of the unreacted components, the sponge was rinsed with distilled water three to four times. The sponge was also dried for three hours at 70˚C in the oven. The sponge that was created was referred to as PVF (Polyvinyl alcohol formaldehyde sponge),

as it is depicted in Figure 1A.

To acquire 1.0 g mass per piece, the dry sponge was cut into square pieces. Next, 75 mL of acetonitrile, 5.0 mL of NH_3, and 0.6 mL of DTMS.H_2O were added to 250 mL RBF, and the mixture was shaken for ten minutes at room temperature. The sponge piece that was previously made was added to the reaction mixture and mixed for 10 minutes. The mixture was then let to sit at 50°C for six hours. After cooling to room temperature, the reaction mixture was filtered. To eliminate unreacted DTMS, the sponge was further cleaned four or five times with acetonitrile and dried for a further twelve hours at 60 °C. In the end, hydrophobic sponge (as seen in Scheme 1) was acquired and used to capture oil from soil samples contaminated with oil.
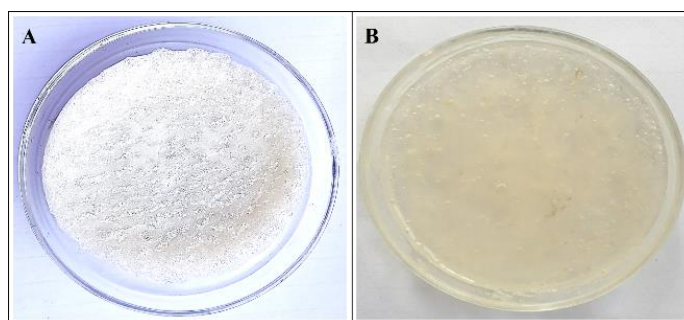
Fig. 1. Photographs of the (A) PVF-sponge and (B) Organic aerogel.

2.3. Synthesis of hydrophobic organic aerogel

In a 250 mL volumetric flask, 1.0 g of chitosan, 20 mL of acetic acid, and 4.0 mL of TEOS were added. The mixture was agitated for ten hours. Stirring for ten hours, the mixture was transferred to a petri dish and let to stand at room temperature for the entire night. The resulting combination was then vacuum freeze-dried and repeatedly cleaned with distilled water. The organic aerogel containing 1.0 g of chitosan was designated as 25-CS, as illustrated in Figure 1B. Lastly, to create the hydrophobic surface of the dried mix aerogel, it was exposed to hexamethyldisilizane (HMDS) vapours for 8 hours at 50–60 °C (as seen in Scheme 2). Because there were no adverse effects and the hydroxy groups in the aerogel and sponge were easily replaced with alkyl groups, the HMDS and DTMS were chosen as surface modifiers. The hydrophobic modified organic aerogel and sponge demonstrated a high porosity, large surface area, and exceptional hydrophobicity [17].

2.4. Characterizations

The prepared aerogel and sponge samples were characterized using a variety of techniques. The surface morphologies of the synthesized aerogel and sponge samples were analysed using SEM, and the chemical compositions were examined using FTIR from Central Instrumentation Lab MM (DU) Mullana, Ambala, India.

2.5. Determining the surface area of the aerogel and prepared sponge samples

The surface areas of the hydrophobic PVF sponge and organic aerogel samples were calculated using the Sears technique [18, 19]. The 1.5 g samples of the aerogel and sponge were taken individually and mixed with 30.0 g of NaCl and 100 mL of water in two different 500 mL beakers. The resulting combinations were whirled for five minutes. Additionally, 0.1 N HCl was added to each combination, making a total volume of 150 mL. Finally, the pH of the mixtures was raised from 4.0 to 9.0 using 0.1 N NaOH. The surface area and required amount of 0.1 N NaOH were calculated using equation (1).

$$A = 32V - 25 \tag{1}$$

where V is the amount of 0.1 N NaOH needed to raise the pH from 4.0 to 9.0 and A is the estimated surface area of the sponge/aerogel samples per gram (m^2/g).

2.6. Pore (void) volume determination

After being immersed in 30.0 mL of distilled water, two distinct 250 mL RBFs holding 2.0 g of the hydrophobic sponge and aerogel each were heated to 100 °C for nearly 30 minutes. The purpose of this process was to remove the absorbed air from the pores of the sponge and aerogel. After thirty minutes of heating, both samples were removed from the RBFs and gently dried by pressing them between the folds of plain filter paper. The dried samples were weighed. Finally, the pore volume was calculated using Equations (2) and (3) [18–20].
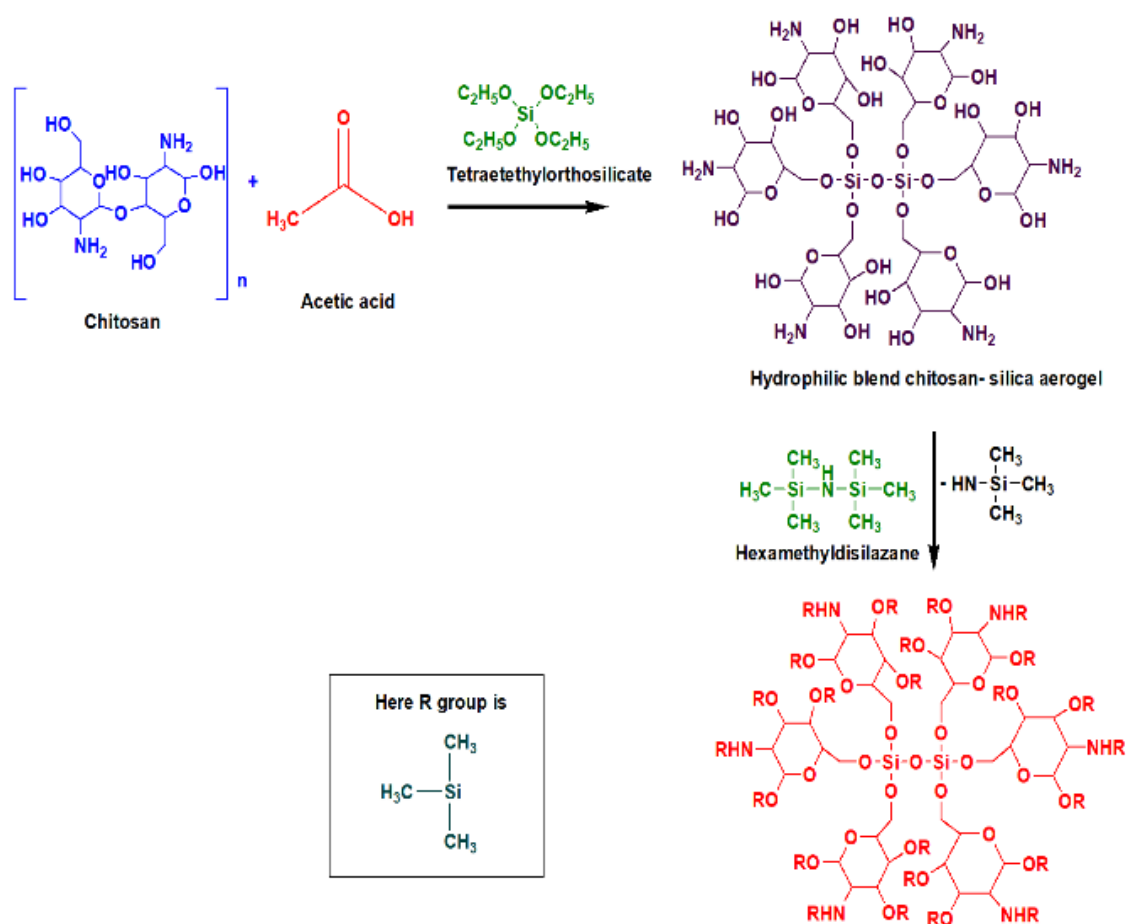
$$\Delta W = W_{final} - W_{initial} \tag{2}$$
$$p_v = \Delta w / p_w \tag{3}$$

Where p_v is the pore volume (cm^3/g), p_w is the water density (g/cm^3), and Δw is the weight (g) difference between the initial ($w_{initial}$) and final (w_{final}) weights of the sponge and aerogel.
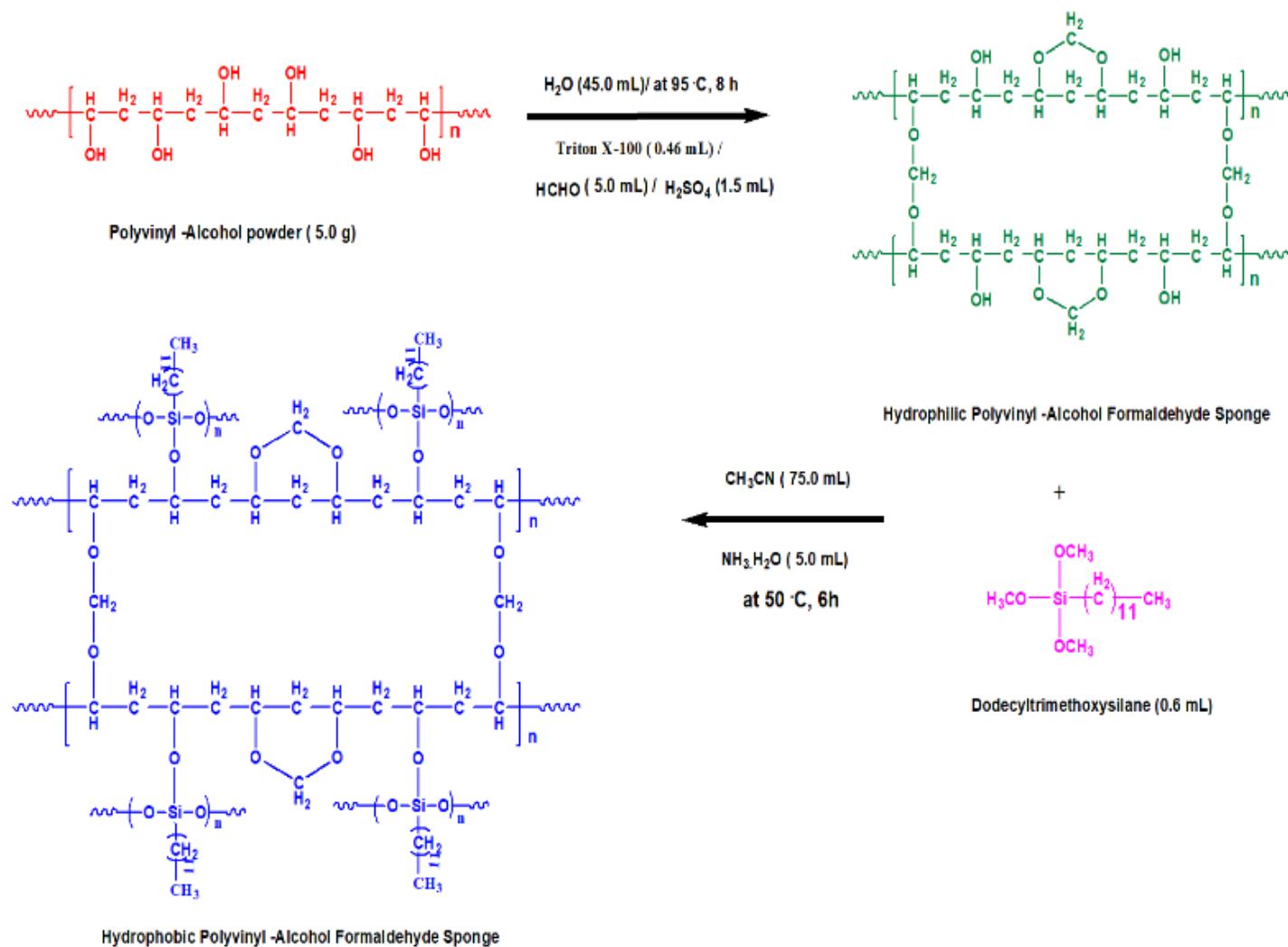
Scheme 1. Reaction Scheme and temperature condition for the design of hydrophobic polyvinyl-alcohol formaldehyde (PVF) sponge.

Scheme 2. Reaction Scheme and temperature condition for the design of hydrophobic organic aerogel.

2.7. Batch adsorption studies

The soil samples were prepared according to the protocol outlined in our earlier article [11]: First, 20 g of diesel-spiked soil sample (DSS, containing 5.0 mL diesel) was agitated for three hours in 500.0 mL single neck RBF using 100 mL deionized water. After filtering the resultant combination, the filtrate was gathered in a different beaker. Finally, 1.0 g of PVF sponge was submerged in the filtrate solution, and the sponge sample was taken out of the beaker after 30 seconds. The sponge was squeezed as much oil as possible with a stainless-steel lemon squeezer, and Equations (4) and (5) were used to determine the percentage oil uptake capacity [12, 21].

$$\%R \ = \ \frac{V_f}{V_i} \ \times \ 100\% \tag{4}$$

$$V_o \ = \ m_f \ - \ m_i \tag{5}$$

where m_f is the sponge's final weight after absorbing oil, m_i is the sponge's initial weight before oil adsorption, and %R is the sponge's percentage of oil absorption capacity. Vi is the initial oil volume in solution. The oil was extracted after the sponge was immersed in the oil-water mixture for nearly seven cycles. The exact same procedure was used with the chitosan-silica organic aerogel.

3. RESULTS AND DISCUSSION

3.1. Characterizations of prepared hydrophobic PVF sponge and organic aerogel

The FTIR spectra of the hydrophobic polyvinyl alcohol formaldehyde sponge and the organic aerogel were displayed in Figure 2. It was noted that the hydrophobic PVF sponge did not exhibit the C-OH peak, which is located about 3300 cm^{-1}. Moreover, the C-H stretching vibration of the alkyl side chain and the Si-O-C vibration are responsible for the peaks observed in PVF sponge at 2960 cm^{-1} and 1050 cm^{-1}, respectively. In mix aerogel, however, a less prominent absorption band was detected around 3400-3200 cm^{-1} for C-OH and Si-OH. Additional stretching vibrations for the mix aerogel's Si-O-Si, C-C, and Si-O-C moieties were measured at 1070 cm^{-1}, 1640-1630 cm^{-1}, and 1090-1030 cm^{-1} [21-23]. The production of the necessary hydrophobic sponge and aerogel was eventually confirmed by the alkyl side chains C-H, which were responsible for the key bands around 790 cm^{-1} and 800 cm^{-1} [24, 25]. As a result, it can be said that FTIR spectra may be a useful method for identifying any alterations or changes made to the generated samples from the original substrates.

Figure 3 displays the findings of a study conducted using scanning electron microscopy (SEM) to examine the morphologies of synthesized materials. The hydrophobic

PVF sponge and the organic aerogel are shown in SEM pictures in Figure 3. The hydrophobic sponge and aerogel surfaces were found to have random roughness and many protrusions, which were crucial for the absorption and retention of oil molecules [18, 21]. Additionally, once the aforementioned materials were hydrophobically treated, the size and quantity of the pores increased, making them more effective oil trappers from samples of oil-polluted soil.

3.2. Surface area and pore volume

The surface area and pore volume of the organic aerogel and PVF sponge were compiled in Table 1. In comparison to hydrophobic PVF sponge, it was found that the hydrophobic organic aerogel had a larger surface area. From organic aerogel to PVF sponge, the surface area reduced from 275.8 to 83.8 m^2/g. Furthermore, after seven cycles, the hydrophobic mix aerogel with an enhanced surface area (275.8 m^2/g) produced an exceptional oil pickup capability of 100% from soil samples contaminated with oil [18, 25].

3.3. Visual observations of oil uptake by hydrophobic PVF sponge

The visual observation of the entire diesel oil cleaning process with a hydrophobic PVF sponge is shown in Figure 4. First, a 1.0 g sponge (Figure 4A) was combined with a 40 mL (5 mL oil and 35 mL water) oil-water mixture at a ratio of 1:7 (Figure 4C).
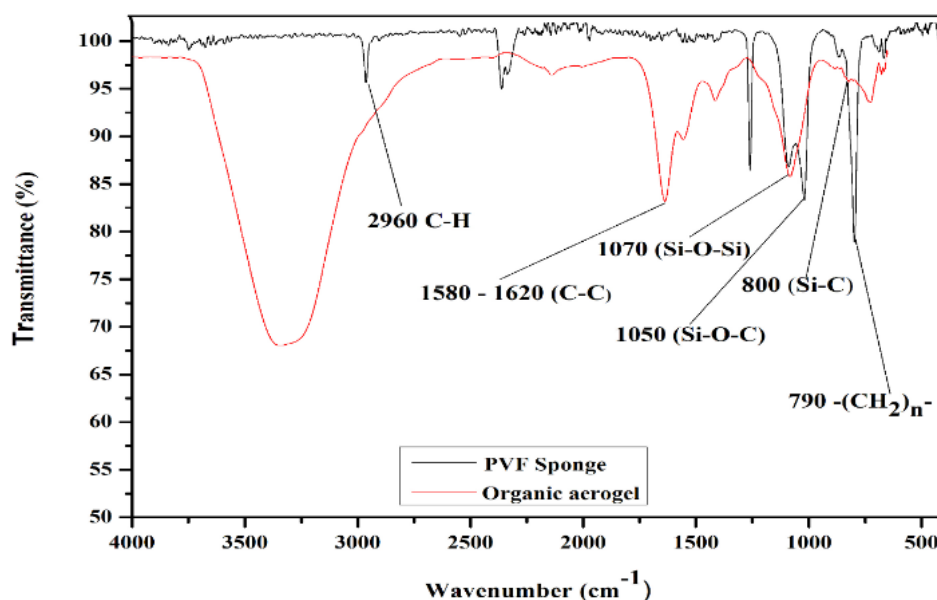

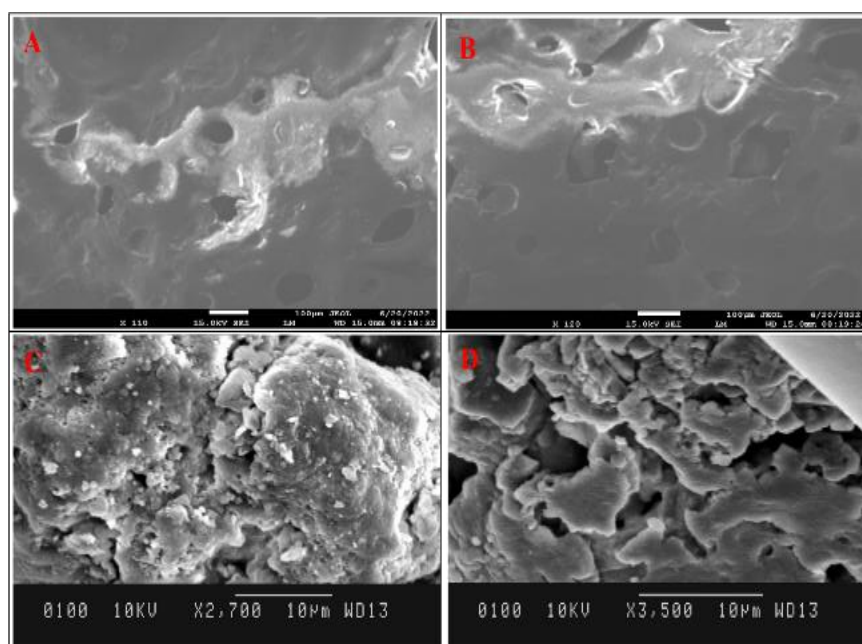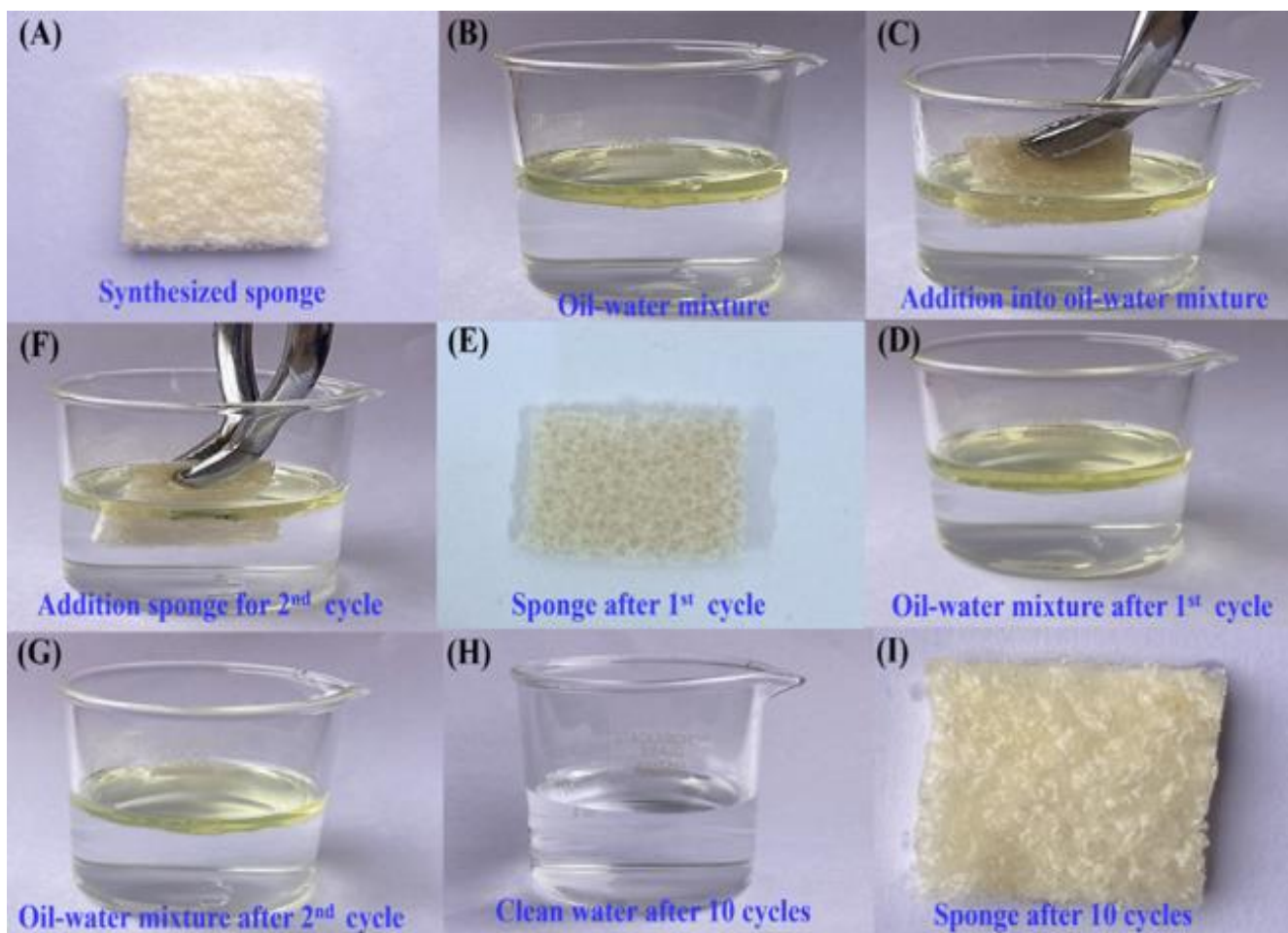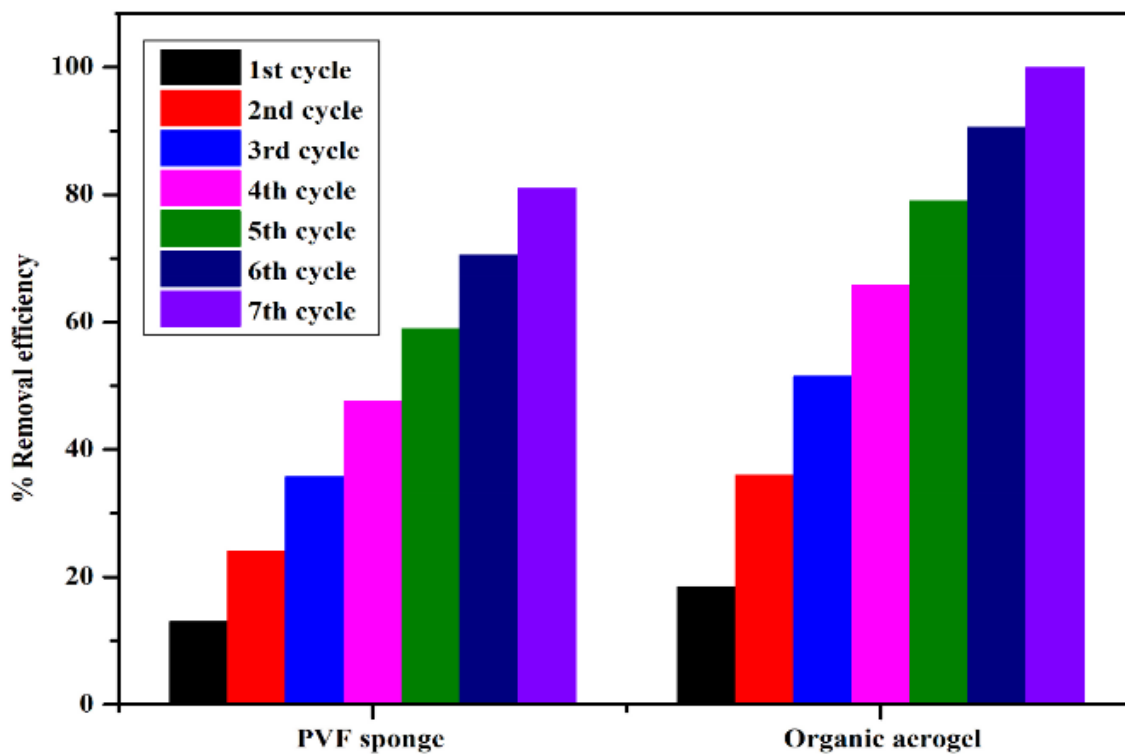**Fig. 2.** FTIR spectra of hydrophobic PVF sponge and Organic aerogel.

Fig. 3. SEM images of the (A and B) PVF-sponge and (C and D) Organic aerogel.

Fig. 4. Photographs taken during oil uptake by synthesized hydrophobic PVF sponge.

Fig. 5. Percentage oil removal efficiency using PVF-sponge and organic aerogel.

Table 1. Surface area and pore volume of the hydrophobic PVF sponge and Organic aerogel.

S. No.	Sample	Surface area (m²/g)	Pore volume (cm³/g)
1	Hydrophobic PVF sponge	83.8	1.21
2	Hydrophobic organic aerogel	275.8	0.413

Table 2. Comparison table of first seven cycles for % diesel oil removal efficiency using PVF sponge and Organic aerogel.

Sr. No.	Number of cycles	% Removal efficiency of diesel oil	
		Organic aerogel	**PVF sponge**
1.	1st	18.5	13.0
2.	2nd	36.0	24.0
3.	3rd	51.5	35.8
4.	4th	65.8	47.6
5.	5th	79.0	59.0
6.	6th	90.5	70.5
7.	7th	100	81.0

Table 3. Comparison of oil uptake capacity by different types of sponges/aerogels.

S. No.	Raw materials	Amount of aerogel/sponge (g)	Number of cycles	Removal capacity	Ref.
1	Polyvinyl alcohol	1.0	35	89.3	[28]
2	Polyurethane	1.0	Dna	25	[29]
3	Chitin	1.0	10	29-58	[30]
4	Melamine	1.0	30	61.0	[31]
5	Catechol	1.0	30	99%	[32]
6	Polyvinyl alcohol	1.0	10	1.8-7.0	[33]
7	Polyvinyl alcohol	1.0	10	4.0	[34]
8	Sodium silicate	1.0	10	4.0	[35]
9	Chitosan-silica	1.0	Dna	14-30	[36]
10	N, N'- methylene bisacrylamide and Graphene oxide (GO)	1.0	10	99.8%	[37]
11	Cellulose acetate	1.0	Dna	112	[38]
12	Raw cotton	1.0	Dna	50-190	[39]
13	Waste newspaper	1.0	Dna	29-51	[40]
14	Cellulose nanofibers	1.0	Dna	80-190	[41]
15	Chitosan-silica	1.0	7.0	100%	Present work
	Polyvinyl Alcohol	1.0	10.0	100%	Present Work

*Dna- Data not available

After 30 seconds, the sponge was removed from the liquid, and its weight was noted. It was discovered that the hydrophobic sponge's weight had increased from 1.0 to 1.61 g. The sponge took up 0.61 g of diesel oil from the oil-water combination (Figure 4D). After the sponge was squeezed, ten repetitions of the same procedure were performed until the oil was completely removed (Figure 4H). In the end, the corresponding photos (Figure 4H and 4I) show that the clean water and oil-absorbed sponge were found. About 100% of the oil content (5 mL) was absorbed by the hydrophobic sponge, indicating the wide applicability of the hydrophobic modification and the precise separation of oil from the oil-water mixture by the sponge [26]. The same procedure was

used with organic aerogel, and after seven cycles, 100% oil adsorption was found.

3.4. Batch adsorption studies

These batch adsorption tests produced some quite intriguing results, as Table 2 and Figure 5 demonstrate. The surface area of the adsorbents is essentially what determines the absorption capacity. The information in Table 1 confirmed the observed results. From mix aerogel to PVF-sponge, it was shown that the absorption capacity decreased with decreasing surface area. Using a combination of organic aerogel and PVF-sponge, the highest percentage absorption capacity of 100% and 81% was achieved in seven cycles. When compared to PVF-sponge, the mix aerogel's surface area was maximal and its absorption capacity was highest [27, 28]. Furthermore, the results are corroborated by the quantity and size of pores seen in SEM images. Table 3 compares the oil absorption capability of various sponge and aerogel types that have been created to far. When compared to previous results found in the literature, it was noted that the current research' results were fairly excellent, providing maximal oil uptake with the fewest number of cycles.

4. CONCLUSIONS

In summary, this study's novel strategy for quickly and efficiently removing oil from contaminated soil samples, which makes use of sponges and self-synthesized organic aerogels represents a substantial advancement in environmental remediation. The hydrophobic properties of aerogel and sponge were verified by the Si-O-C bond peaks seen in FTIR spectra. The SEM pictures revealed a randomly rough surface that was very porous, increasing both the volume and size of the pores. Furthermore, the organic aerogel had the largest surface area (275.8 m^2g^{-1}) when the surface area was determined using the Sears method. After seven cycles of batch testing, it was discovered that organic aerogel absorbed 19% more oil from polluted soil samples than sponge did. These materials' exceptional capacity to quickly absorb and encapsulate oil pollutants demonstrates their promise as an environmentally responsible and long-lasting remedy for soil contamination and oil spills. These materials are not only highly effective at absorbing oil, but they can also be easily synthesized and reused, which makes them a cost-effective option for reducing the negative effects of oil pollution. Additionally, this study emphasizes the value of interdisciplinary cooperation by bridging the environmental engineering and materials science gaps and providing workable answers to urgent ecological problems. Research like these give us hope and inspiration as we struggle to address the growing risks posed by climate change and environmental degradation. Self-synthesized organic aerogels and sponges have the potential to transform environmental remediation by providing a more rapid, effective, and sustainable way to rebuild ecosystems and soil.

This research opens the door to a more sustainable, brighter future in the face of rising oil contamination challenges. We sincerely hope that the encouraging findings shared here will inspire more study and advancement in this area, which will finally result in useful applications that will significantly improve the environment and preserve the planet's health for future generations.

5. PROSPECTIVE VIEW

In the future, there are numerous directions this topic could go for research and development:

1. Optimization: To further increase the oil absorbing capacity and efficiency of organic aerogels and sponges, future research can concentrate on optimizing the synthesis process of these materials. This can entail modifying the materials to accommodate particular kinds of oil pollutants.
2. Long-Term Effects: It's important to look into how employing these products for soil remediation will affect the environment down the road. It's critical to evaluate their long-term stability, robustness, and potential effects on ecosystems and soil quality.
3. Field Testing: To confirm the practical viability of these materials, a shift from laboratory investigations to field trials is necessary. Applications in the real world and monitoring can offer insightful information on how they function in various environmental settings.
4. Cost-Effectiveness: Before employing self-synthesised organic aerogels and sponges for oil remediation on a broader scale, it is imperative to assess their cost-effectiveness. There is interest in lowering production costs and determining their viability economically.
5. Combination with Other Remediation Techniques: Investigating the combination of organic aerogels and sponges with chemical treatments or bioremediation can result in synergistic approaches that improve the effectiveness of soil remediation even further.

Using self-synthesized organic aerogels and sponges, this study concludes with a promising method for the quick and efficient removal of oil pollutants from soil. Even if the current results are promising, more investigation and development are required to fully realize the potential of these materials and tackle the problems associated with oil contamination in soil on a larger scale.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests.

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