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Article

SiO_2/PDMS modified porous systems for oil removal: reuse cycles studied

Modificación de sistemas porosos con SiO_2/PDMS como removedores de aceite: estudio de ciclos de reúso

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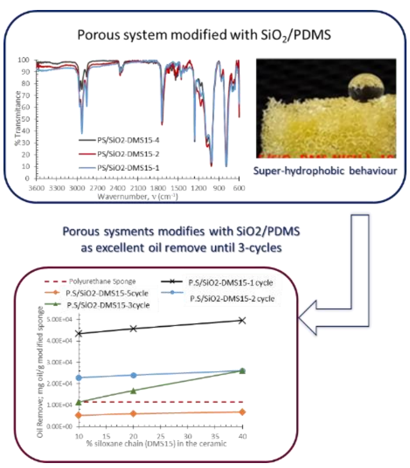
Abstract

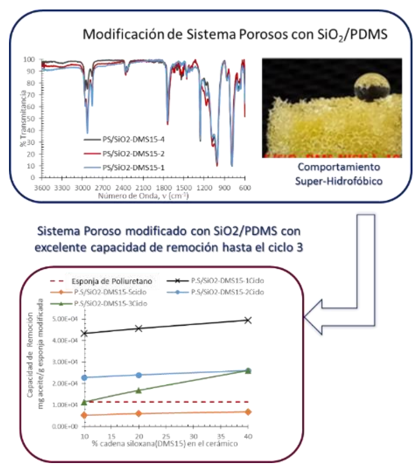
Currently, water pollution is a global problem that must be addressed; since, added to the aridity cycles, they have caused a shortage of natural water resources. Therefore, improving wastewater treatment methods is a global research topic that needs to be developed. Among the contaminants present in wastewater (industry and domestic) are “oily substances” which are dispersed in the water, making their removal difficult. Therefore, this project seeks to determine the reuse capacity for an oily substance removal system designed from the modification of a porous medium with a hydrophobic ceramic (SiO_2/PDMS) that contains in the structure of the siloxane chain the methyl functional group (-CH_3). To do this, the liquid-liquid extraction of the oil removed from the sponge/ceramic was carried out using solvents such as: hexane and THF; determining the numbers of use cycles, identifying by infrared spectroscopy the modifications in the modified sponge after each use cycle.



SiO2/PDMS, Oil remotion, Reuse-cycles

Resumen

En la actualidad, la contaminación del agua es un problema mundial que debe ser atendido; ya que sumado a los ciclos aridez han provocado una escases del recurso hídrico natural. Por lo que mejorar los métodos de tratamiento de aguas residuales es un tema de investigación mundial necesario por desarrollar. Entre los contaminantes presentes en las aguas residuales (industria y doméstica) se encuentra las “sustancias oleosas” las cuales se dispersan en el agua dificultando su remoción. Por lo que, en este proyecto se busca determinar la capacidad de re-uso para un sistema de remoción de sustancia oleosas diseñado a partir de la modificación de un medio poroso con un cerámico hidrofóbico (SiO_2/PDMS) que contiene en la estructura de la cadena siloxana el grupo funcional metilo (-CH_3). Para ello, se realizó la extracción líquido-líquido del aceite removido en la esponja/cerámico empleando disolventes tales como: hexano y THF; determinando los números de ciclos de uso identificando por espectroscopia de infrarrojo las modificaciones en la esponja modificada después de cada ciclo de uso.



SiO2/PDMS, Remoción de aceite, Ciclos de reúso

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Introduction

The use of adsorbent systems as a means for the removal of contaminants represents a highly efficient alternative, allowing for the elimination of various types of contaminants through the control of the interactions between the sorbate and the adsorbent. In this regard, mesoporous silica is an inorganic solid with a structured pore network and high adsorption capacity (Technologies, 2024; Seisenbaeya G.A; et.al 2021; Ulfa M, et.al 2022; Flores D, et.al, 2022). It has been demonstrated that a range of contaminants, including heavy metals (Grozdov D, et.al 2023; Zhu W, et.al 2017; Nicola R, et. al 2020), dyes chlorinated compounds (Quin Q, et.al, 2012),, and pesticides (Kong X.P, et.al 2021), can be efficiently adsorbed on these materials, with removal efficiencies ranging from good to excellent (Palomino J.M, 2014, Wu Z, et.al, 2011).

On the other hand, silica offers the advantage of straightforward functionalization of the surface. Figure 1 demonstrates that, through reactions with the silanol groups present on the surface, condensation reactions can be conducted with alkylalkoxysilanes, enable the addition of diverse molecular types (Ghosh S, et. al, 2013; Tian J, et. al. 2015). These groups include amino, thiol, and alkyl groups, as well as proteins, sugars, enzymes, and molecular recognizers (Kucinski K, et. al 2018). As a result, they can be used as selective adsorbents in a variety of fields, including metallurgy (Salazar-Hernández M, 2024), pharmaceuticals (jaafar J.A, et. al, 2019; Kirla H, et. al, 2023), and the environment (Xu P, et.al, 2018; Song Y, et.al, 2019; Abdulazeez I, et. al, 2023).

Box 1

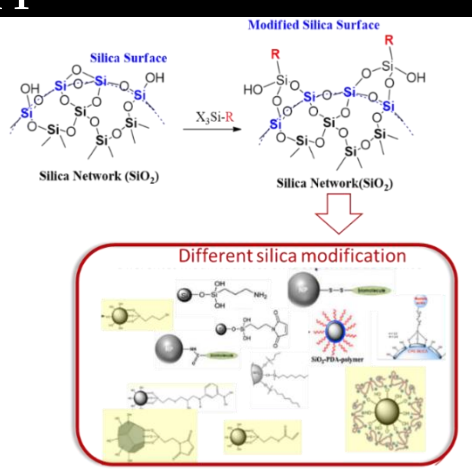

Figure 1
Modification of the silica surface with different functional groups

Table 1 provides an overview of the environmental applications of silica modified with alkyl (–R) groups, which impart hydrophobic characteristics. These materials have been employed for the removal of a range of pollutants, including Cu(I), Cd (II), Pb(II), Active Red X-3B, and rhodamine B, as well as organic solvents such as phenol, toluene, trichloroethylene, and chlorobenzene, as well as oil and fatty substances. To achieve the removal of contaminants, hydrophobic silica is deposited on matrices such as cellulose, chitosan, carbon nanotubes, graphene oxide, and others (Salazar-Hernández C, et.al, 2021; Gómez-López R.V, et.al 2022).

Box 2

Table 1
Environmental applications of hydrophobic silica (Gómez-López R.V, 2022)

Applications	Silica deposition matrix	Adsorbate	Removal
Removal of metal and organic contaminants	Cellulose-silica aerogel	Cu (I)	801 mg/g
	RC-base aerogel	Cd(II)	100.16 mg/g
		Pb(II)	152.23 mg/g
	Chitosan	Pb(II)	102.03 mg/g
	Cellulose-chitosan	Active red X-3B	100%
	CNT and chitosan	Chipton	227.3 mg/g
	Graphene Oxide	Fenol	99%
	cellulose	Rhodamine B	97%
Organic Solvents and oil removal	CF_3 Functionalized	oil	Absorbs 14 times more oil than Aerogel weighs
	Reduced Graphene Oxide	Fatty substance	4517-14728%w
	Cellulose-silica hybrid	Fatty substance	24.8 g/g
	TFPTMOS y TMOS	Toluene	833 mg/g
		Ethanol	458 mg/g
		Chlorobenzene	11890 mg/g
		Trichloroethylene	1935 mg/g

Source: Own elaboration

In this context, Salazar-Hernandez et al. have developed a modification of the silica surface with different organic functional groups, including methyl, octyl, and polysiloxane (PDMS), which has been observed to increase the hydrophobic character as follows: $SiO_2 < SiO_2–CH_3 < SiO_2$–octyl $< SiO_2$–PDMS.

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The results showed that the hydrophobic character of the modified silica serves to control the oil removal capacity through flocculation, resulting in a maximum for the SiO_2/PDMS system of 4 g oil/g SiO_2–PDMS (Gómez-López R.V, et. al, 2022). The feasibility of using a porous system (polyurethane sponge) as a removal system for oily substances was determined by modifying it with the SiO_2/PDMS ceramic. Figure 2 illustrates the contaminant removal capacity, demonstrating a removal capacity of 10 g of oil per m² of treated sponge, which is equivalent to 12.25 g oil/g of modified sponge (Gómez-López, R.V, et. al, 2022; Feng X, et. al 2024).

Box 3

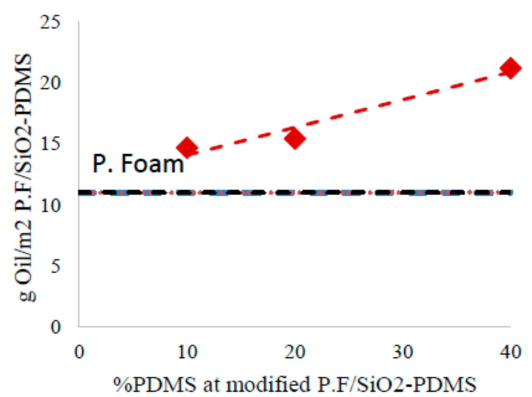

Figure 2
Oil removal capacity for P.F/SiO_2-PDMS
Source: Take from Gómez-López R.V, et.al 2022

Thus, this study aims to present the removal capacity of foam modified with ceramic in different contaminant removal-extraction cycles.

Experimental procedure

Synthesis of SiO_2/PDMS and the modification of polyurethane

The silica modification was conducted via co-condensation, as previously reported by Salazar–Hernandez et al 2021. The polymerization of TEOS (Aldrich; 99%) is conducted by magnetic stirring for 30 minutes at 50 °C, with the addition of PDMS (Gelest) and DBTL as a polycondensation catalyst. Table 2 provides a detailed account of the concentrations of PDMS used in the silica modification process. The polyurethane sponge (5 mm x 3 mm x 2 mm) was subjected to impregnation, after which it was dried at 50°C for 24 hours.

Box 4

Table 2
Amounts of TEOS/PDMS used for silica modification.

	TEOS (g)	PDMS (g)
SiO_2/DMS15-1	10	1
SiO_2/DMS15-2	10	2
SiO_2/DMS15-4	10	4

Characterization of the ceramic-modified sponge

ATR-FT. Attenuated total reflection Fourier transform spectroscopy (ATR-FT) was employed to obtain the infrared spectra. This was conducted using a Nicolet iS10 spectrometer from Thermo Scientific, with an average of 16 scans acquired across a spectral window of 4000–600 cm^{-1} and a resolution of 4 cm^{-1}.

Hidrophobicity-Contact Angle. The contact angle for the sponge and sponge-modified was determined using a drop of distilled water from L. The image was captured with a conventional cell camera and the contact angle was measured with the free software IC-Measure, with the horizontal base serving as the reference point.

Oil removal test. The oil absorption process was initiated by adding 20 mL of water with red vegetable dye at a concentration of 0.001% (vol./vol.) to a beaker containing 2 mL of vegetable oil (commercial vegetable oil). This mixture was prepared as an emulsion in synthetic water to facilitate experimentation. The modified sponge was then introduced into the emulsion to determine the quantity of oil removed, with measurements taken with a test tube.

Oil extraction procedure. The organic solvent was added for oil extraction, with the extraction cycles conducted with constant agitation for five minutes in an orbital shaker. Thereafter, the solution was allowed to drain, and it was subsequently dried in an oven at 80°C for 24 hours to eliminate the impregnated solvent. Subsequently, the structural modification of the material was assessed through the evidence of its infrared spectra.

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Results and discussions

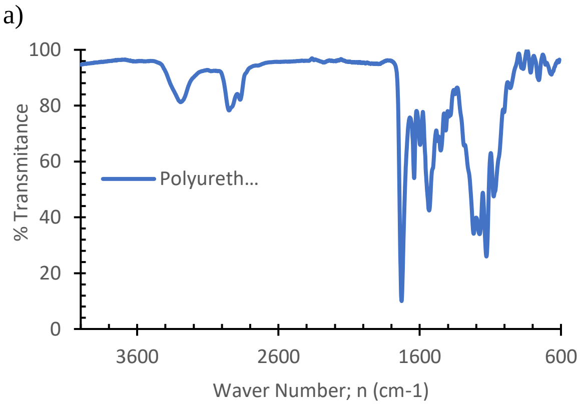
Sponge modified with SiO_2/PDMS ceramics

Figure 3a shows the infrared spectrum for the unmodified sponge. It shows the characteristic functional groups for polyurethane at 1650 cm^{-1} and 1708 cm^{-1}. It also shows the N–H at 3294 cm^{-1} and the C–O–C at 1094 cm^{-1}. C at 1094 cm^{-1}, C–H at 2966–2867 and 1440 cm^{-1}, C–N at 1020–1220 cm^{-1}, C=C at 1603, 1580 cm^{-1}. Figure 3b shows that this material is hydrophilic, meaning it wets out easily.

Upon modification with the ceramic (Figure 3c), the signals of the impregnated foam are identified. At 1100 and 790 cm^{-1}, the Si–O–Si network is observed, while at 1200 cm^{-1}, the Si–C group of the siloxane chain is observed at 900 cm^{-1}, showing as an intense and sharp band. Additionally, the Si-O-Si group of the siloxane chain is discernible at this same wavelength, revealing a distinct and prominent peak. Finally, the C–H of the $–CH_3$ is evident at 2900 cm^{-1}. On the other hand, it was observed that the sponge underwent further modification as the DMS15 content in the ceramic increased. Furthermore, the ceramic induces a transition from hydrophilic to hydrophobic behavior in the sponge (Figure 3d), as evidenced by a modification of the contact angle to a value of 136–140° in accordance with the increase in the siloxane chain length within the silica network. The results suggest an excellent modification of polyurethane sponge with ceramic SiO2/PDMS according to previous reports of this hybrid ceramic (Feng X, et. al, 2024; Chen K, et. al, 2024; Xu S, et. al 2024)

Box 5

a)



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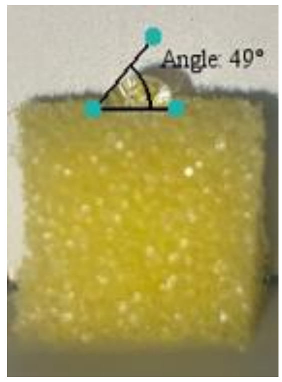

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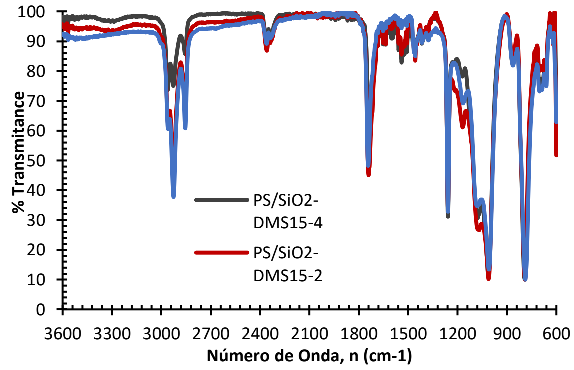

d)

Figure 3
Characterization of the polyurethane sponge (a) infrared spectrum before modification (b) contact angle before modification (d) infrared spectrum modified with the ceramic (e) contact angle after modification with ceramic

Figure 4 shows the effect of siloxane chain content on the hydrophobicity of the material, as indicated by the contact angle (θ). The data demonstrate that an increase in the DMS15 content in the ceramic resulted in a corresponding increase in the contact angle. Specifically, the ceramic with 40% DMS15 exhibited a contact angle of 141°, while the ceramic with 10% DMS15 displayed a contact angle of 136.75°. Conversely, the data indicates that the ceramics exhibit superhydrophobic characteristics (Zhou E, et. al, 2024), as evidenced by a contact angle exceeding 120° (Danish M, 2022).

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Box 6

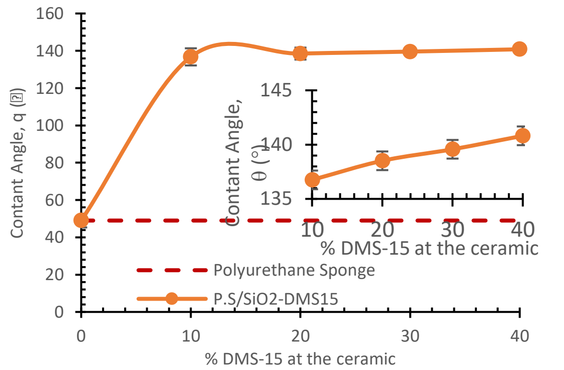

Figure 4
Effect of siloxane chain content (DMS15) in the silica network on hydrophobic behavior

Oil Removal Tests

Reuse Cycles

Figure 5 shows how well the unmodified sponge, and the sponge modified with different siloxane chain contents (DMS15) remove oil. The unmodified sponge can only be used once because the oil gets in between its pores. To choose the best solvent for extraction, we tested three common solvents: kerosene, THF, and hexane. We found that hexane was the most effective at removing the oil, with a recovery percentage of 45-50%. Hermogenes et al. 2010 have found this organic solvent works well for extraction, even though it has a moderate recovery percentage.

Box 7

Table 3
Oil extraction capacity in water with organic solvents

	% Oil recovery
Kerosene	20
THF	40
Hexane	45–50

The results demonstrated that the higher the DMS15 content in the ceramic, the greater the removal capacity. Table 4 shows the increase in the removal of the modified sponges in each cycle. In the first cycle, for example, the removal capacity was 1.7 times greater for the sponge modified with the ceramic containing 10% of the siloxane chain (DMS15) than for the control sponge.

In the initial cycle, the sponge modified with the ceramic containing 10% of the siloxane chain (DMS15) exhibited an increase of 3.8 times, while the DMS15 content was increased to 40% in the subsequent cycle, resulting in a 4.5 increase.

Box 8

Table 4
Oil removal capacity of the modified sponge

	Removal Cycle			
	1	2	3	5
Polyurethane Sponge	1	---	---	---
SP/SiO2-DMS15-1	3.8	2	1	0.46
SP/SiO2-DMS15-2	4	2.10	1.52	0.52
SP/SiO2-DMS15-4	4.5	2.3	2.28	0.62

Box 9

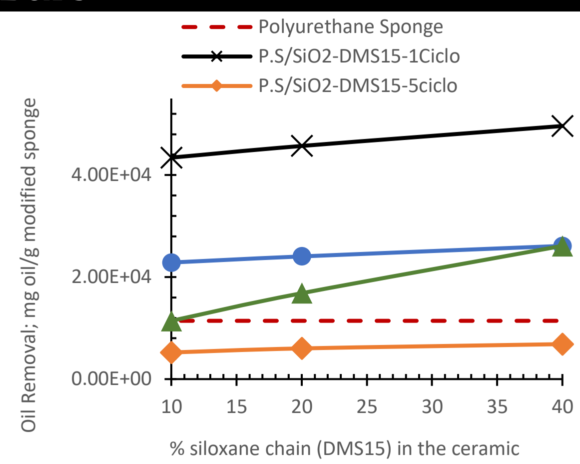

Figure 5
Oil removal capacity of the SiO2/DMS15 modified sponge with varying siloxane chain content.

Figure 6 shows the corresponding spectra for the modified sponge following the initial and second removal cycles. In the second removal cycle, the sponge exhibited a notable loss in its adsorption capacity, reaching approximately 50% as observed in Figure 6b. Following the initial removal cycle, the ceramic remained unaltered in its structural composition, with the exception of a reduction in the intensity of the bands at 1200 cm^{-1} (Si–C). The spectra also show a decrease in the intensity of the Si–C band at 1200 cm^{-1}, which is attributed to the removal of the siloxane chain, and an increase in the intensity of the Si-O-Si band at 1100 cm^{-1}, which is associated with the formation of a silica network. Additionally, there is an observed increase in the intensity of the C–H band between 2800 and 2900 cm^{-1}, which may suggest the presence of a thin layer of oil that is not fully extracted from the system. This could explain the observed decrease in the observed removal capacity.

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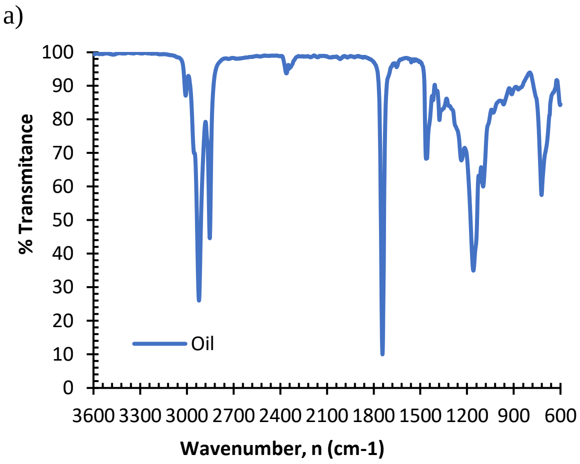
In the third cycle, the removal capacity decreases by approximately 70%. According to the infrared spectrum after the second cycle (Figure 6c), the oil layer retained in the ceramic sponge is predominant, with the signals corresponding to the ceramic (1200 cm^{-1} and 1100 cm^{-1}) observed with very low intensity. In subsequent cycles, the quantity of oil is markedly elevated, and the presence of ceramic is no longer evident.

Hexane is inadequate for the complete extraction of the oily substance. Consequently, a residual layer is retained from the initial cycle, which increases with each subsequent cycle, thereby reducing the removal capacity. The functional use of the removal system is limited to up to five cycles.

Box 10

a)



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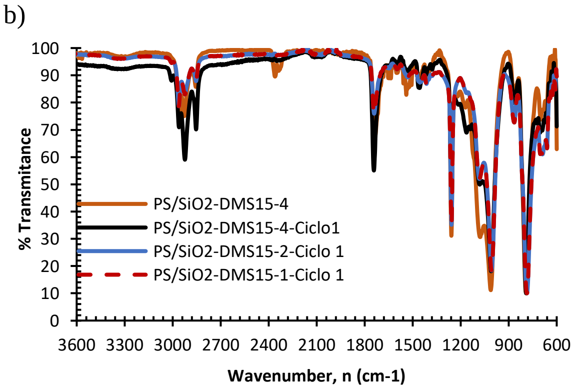
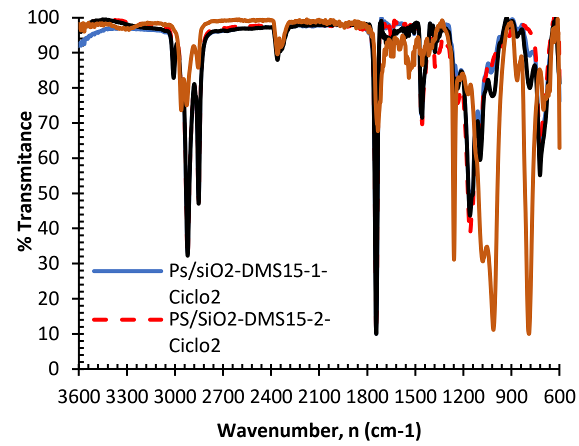


c)



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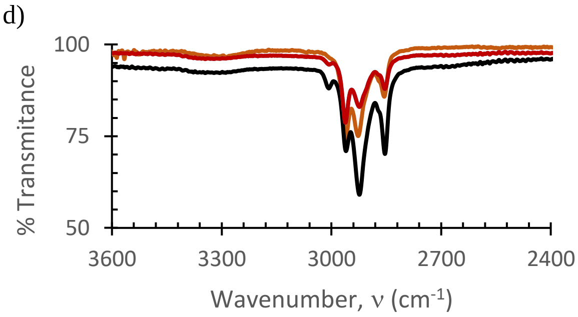

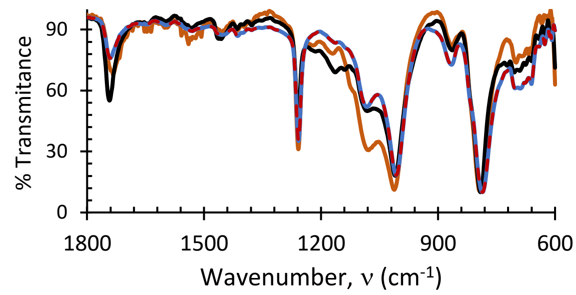

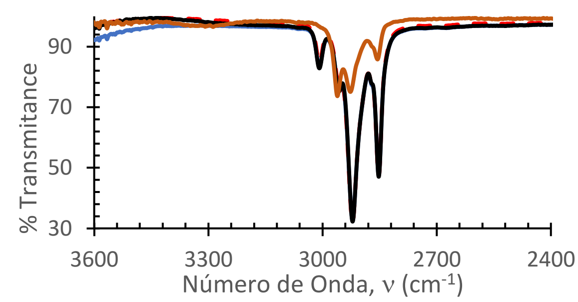

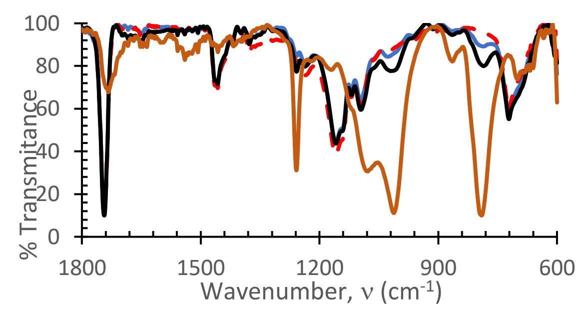

Figure 8

Modification of PS/SiO_2–DMS15 after each removal cycle (a) vegetable oil (b) first cycle (b) second cycle

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Conclusions

The modification of adsorption systems, such as the polyurethane sponge, with hydrophobic ceramics (SiO_2/PDMS-CH_3), has been demonstrated to enhance the retention capacity of oily agents, including oil. Nevertheless, a significant challenge inherent to any method of contaminant removal is the effective extraction and recovery of the contaminant from the extractant medium.

Despite the use of an organic solvent related to oil (hexane), the recovery of the dispersed oil in water was found to be only 45–50%. Consequently, based on the infrared spectra obtained for the PS/SiO_2–DMS15–CH_3 from the initial cycle, a minimum layer of oil is retained, and its concentration increases with each subsequent cycle. This results in a gradual decrease in the contaminant removal capacity.

The optimal number of cycles for use-reuse of the PS/SiO_2–DMS15–CH_3 system is five. This is based on the observation that under the extraction conditions used, the concentration of oil in water is significantly higher than that found in wastewater. Consequently, the time of use and application of these systems can be highly effective and low cost.

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Statements & declarations

Consent to participate and Consent for publication

The authors express their approval to participate and publish this work in ECORFAN Journal

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

All authors contributed to the development and revision of the manuscript; CSH and MSH (conceptualization, interpretation and analysis date; writing and financial support); JMMM (interpretation and analysis date and methodology), MRLR (interpretation and acquisitions date). All authors read and approved the final manuscript.

Availability of data and materials

Indicate the availability of the data obtained in this research.

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Abbreviations

SiO_2	Silica ceramic
PDMS	Polydimethylsiloxane
SiO_2/PDMS	Silica ceramic modified with polydimethylsiloxane
SiO_2/DMS15-1	Silica ceramic modified with 10 % weight polydimethylsiloxane
SiO_2/DMS15-2	Silica ceramic modified with 20 % weight polydimethylsiloxane
SiO_2/DMS15-4	Silica ceramic modified with 40 % weight polydimethylsiloxane
P.S	Polyurethane sponge
	Contact angle

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Antecedents

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