

Study on the Mechanism of Rapid oil-water separation by Fe₃O₄@PMMA@PDMS intelligent super hydrophobic micro/Nano robot in Indian context

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Abstract

The development of a new superhydrophobic magnetic melamine sponge (PDMS@Fe₃O₄/MS) for effective oil-water separation is presented in the paper. A modified melamine sponge, polydopamine sheath, and nano-Fe₃O₄ particles combine to form a unique superhydrophobic advanced material with a liquid contact angle of 150.9°. The modified sponge exposes a liquid, which is uniquely revealed. Scanning electron microscopy (SEM) characteristics show structural changes throughout the process. X-ray diffraction (XRD) analysis shows that the presence of nano-Fe₃O₄ does not cause the melamine sponge to produce distinctive diffraction peaks and also confirms that the sponge is well coated with PDMS. According to Fourier transform infrared spectroscopy (FTIR), oxygenated functional reducing groups appear on the PDMS@Fe₃O₄/MS surface, enhancing its hydrophobicity and lipophilicity. The study of oil adsorption capacity shows that PDMS@Fe₃O₄/MS can absorb various oils. Due to the three-dimensional porosity of the sponge and the combined water repellent properties of the PDMS, the material shows remarkable oil and water separation efficiency up to 99.34%. Performance tests show that the material can go through several cycles of oil absorption and deoiling, but the rate of absorption and wear gradually decreases. The nano-Fe₃O₄-enhanced magnetic reactivity of the sponge allows magnetic fields to be used for efficient handling and recycling.

Keywords: *Superhydrophobic, Melamine Sponge, Oil-Water Separation, Nano-Fe₃O₄, Adsorption Capacity and, Magnetic Manipulation*

Introduction

Large-scale industrial production and oil spills from everyday life are the driving forces behind the rapid pace of modern socio-economic development. Meanwhile, marine oil spills are increasing. In the case of oil extraction, this scenario will create important environmental issues. People's awareness of the need to preserve the natural environment has given rise to the need to progressively address oily wastewater and marine oil spills. A very important step towards environmental protection around protection and reducing waste is to use oil-water separation, chemical waste separation, and biological separation. But in the face of increasing oil pollution issues, these processes have shown shortcomings: low separation, limited recyclability, inadequate environmental protection, and therefore limited innovation in excess water. To ensure

efficiency and environmentally friendly oil and water separation, a new kind of superhydrophobic material must be developed.

The advantages of the dip coating process are its simplicity, efficiency, affordability, and environmental protection. In recent years, this thermal method has been used for the preparation of non-aqueous and oil-absorbing materials. The high porosity and inexpensive cost of synthetic oil-absorbing sponges are also increasing interest in their oil extraction properties. As a result, there is considerable interest in an underwater method of sponge absorption of excess water. Modified polyurethane sponges have been widely used for oil-water separation.

In accordance with the polyurethane sponge, Lu fabricated a superhydrophobic PDMS. However, the procedure involves the use of chromic acid solution, which is hazardous for nature as well as individuals. The polyurethane sponge that is superhydrophobic was developed by Yang. Although it requires a tedious and expensive manufacturing procedure, its applicability is limited due to this weakness. Disadvantages of polyurethane sponges also include poor open-cell structure and a lack of flammability. When the polyurethane sponge burned, it released toxic fumes. Due to these two drawbacks, the polyurethane sponge fails to meet the goals of environmental protection, high oil and water separation, and handling efficiency. Formaldehyde-melamine copolymer fabrication followed by foaming results in a low-density melamine sponge (MS). With a porosity of more than 99%, it has a three-dimensional pore structure and releases no harmful chemicals when burned. It has unique environmental protection and chemical-stabilising properties. Melamine sponges are great adsorption carriers due to their exceptional chemical stability and environmental protection properties. Melamine sponges are amphibious by nature; this must be emphasized. Certainly, modifying the melamine sponge to make it waterproof is the only way to use it for separating oil and water.

Given the aforementioned advantages, this article decides to use MS as a highly absorbent sponge. Dopamine hydrochloride undergoes oxidative polymerization to form polydopamine sheaths. Because of its high adhesion, polydopamine can act as a "glue" to hold PDMS and nano-Fe₃O₄ together during the preparation phase. After completing the oil-water separation, it was decided to add nano-Fe₃O₄ to enable the sponge to be recycled using a magnetic field. Recycling has the potential to save costs. MS can have unusual hydration, as in the case of PDMS. Finally, we obtain a superhydrophobic melamine sponge (PDMS@Fe₃O₄/MS) by the dipping method. The preparation method is more suitable for use and popularisation because it is cheaper and easier to use.

Objectives of the study

- To develop superhydrophobic melamine sponges.

- To utilise scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR) to investigate structural and chemical changes in the sponge.
- To check the efficiency of the product, check the absorbency of the oils, and calculate the efficiency of oil-water separation.
- To check the performance of the product can be tested for reusability through repeated oil drying and deoiling.
- To investigate whether magnetic manipulation can improve oil-water separation.

Need of the study

The study has highlighted the urgent need for innovative approaches to the problems of oil pollution and oily wastewater treatment. The research seeks to close this gap by developing a superhydrophobic magnetic melamine sponge (PDMS@Fe₃O₄/MS), which demonstrates the limitations of traditional methods in terms of efficient oil and water separation, recycling, and material environmental protection due to industrial activities and oil spills. PDMS is modified by adding nano-Fe₃O₄ to polydopamine sheaths, resulting in improved water absorption. Such improvements are necessary to address the shortcomings of current separation methods, promoting environmentally friendly practices to encourage our environment, offering a flexible, reusable oil-water separation solution that is environmentally sensitive and benign oil-water separation techniques, and showing promise to be widely adopted in dealing with the immediate problems of oil pollution and water pollution.

Materials and Procedures for Experiments

Melamine sponge (Indus Valley Equipment Company, Ltd., Mumbai, India); nano-Fe₃O₄ (20nm 99.0% metal base, Sigma-Aldrich Pharmaceutical Co., Ltd., Bangalore, India); absolute ethanol (Merck Life Sciences India Pvt. Ltd., Mumbai, India); dopamine hydrochloride (HiMedia Laboratory Pvt. Ltd., Mumbai, India); cyclohexane (AR, Thermo Fisher Science India Pvt. Ltd., Mumbai, India); PDMS; Chemical Therapeutics. Instrumentation and precision electronic balance of the ES type; contact angle measuring device of the SDC-200S type (Uni-Tech Instruments Pvt. Ltd., Mumbai, India); BILON3-120A ultrasonic purifier (Crest Analyzers Pvt. Ltd., Mumbai, India); HJ-1 type magnetic heaters and vibrators (Labtech Instruments Pvt. Ltd., Kolkata, India); DZF-6030 vacuum furnace (National Instruments India Pvt. Ltd., Bangalore, India); Apreo-2 field radiation detection electron microscope (JEOL Ltd., New Delhi, India);

Setting up the PDMS at Fe₃O₄/MS

MS surface preparation:

Two cleanings of MS were performed using deionized water and absolute ethanol. After that, air-dry it for six hours at 45 °C in an oven. When it has dried, remove it. The cleaned MS may be shown in Figure 1.

Figure 1. (a) MS and (b) PDMS@Fe₃O₄/MS.



The MS skeleton is coated with polydopamine

Weighing 240 mg of dopamine hydrochloride powder and dissolving it in 60 mL of water will yield a dopamine hydrochloride solution with a concentration of 4 mg/mL that is well absorbed into the MS. Dip the sponge into the solution. Then mix with a magnetic stirrer for an hour. Remove the MS and leave it in the atmosphere all day to permit full oxidation and polymerization of the dopamine hydrochloride.

Nano-Adhesion Sponge-Fe₃O₄

To prepare a suspension, weigh 0.1 g of nano-Fe₃O₄ and dissolve it in a certain volume of ethanol solution. After incubation, the sponge should be ultrasonic-sprayed for one hour. Following sonication, the sponge is dried at 85 °C in a temperature-controlled oven. Remove the sponge after it dries so you can use it later.

The superhydrophobicity of the sponge is treated with PDMS.

Mix manually 1 g of PDMS, 20 mL of cyclohexane solution, and 0.1 g of hardener for one minute. Then, the sponge is fully immersed in the formulation, placed in a drying box at a steady temperature, and dried for 12 hours at 85 °C. Remove the PDMS@Fe₃O₄/MS and cool to room temperature before dipping three times several times with 100% ethanol and cleaning. After washing, dry, then obtain PDMS@Fe₃O₄/MS, as seen in FIG.

Adsorption capacity calculation for PDMS@Fe3O4/MS

Oil and water separation efficiency of PDMS@Fe3O4/MS is greatly influenced by the oil absorption capacity. We chose to investigate the adsorption capacity of kerosene, diesel oil, and maize germ oil to ascertain PDMS@Fe3O4/MS's maximum oil adsorption capability. Weigh ten grammes of each oil and add PDMS@Fe3O4/MS. After drying for a minute, remove the sponge. The absorbance calculation procedure is as follows:

$$Q_f = \frac{m_o - m}{m} \quad (1)$$

Formula (1) states that the mass of PDMS@Fe3O4/MS (ms) is indicated in g, the absorbance (Qf) is indicated in g/g, and the mass (ms0) is the mass of PDMS@Fe3O4/MS after the extraction of oil.

Calculating the PDMS@Fe3O4/MS oil-water separation efficiency

The efficiency of the modified sponge in oily wastewater treatment depends largely on the efficiency of separating oil and water. Fill three oil-water combinations with PDMS@Fe3O4/MS and perform the oil-water separation test. Finally, the oil-water separation efficiency is calculated using the weighting method. The following formula (2) represents the calculation of oil and water separation efficiency.

$$R = \frac{C_1 - C_2}{C} \times 100\% \quad (2)$$

Formula (2): where C is the primary concentration and g is the efficiency of oil and water separation during the oil phase. The volume (C1) of the combination of oil and water is the volume (g) before separation, and the volume (C2) is the volume (g) after separation.

Results and discussion

Analysis and interpretation of the basic modified sponge

Image analysis of MS, Fe3O4/MS, and PDMS@Fe3O4/MS 1 μm scanning electron micrographs is shown in Figure 3a–c. The MS bone surface is smooth and flat, as can be seen by looking at Fig. (a). However, due to the high presence of hydrophilic amino groups in MS, it is not to be used to separate oil and water. Some chemicals are clearly bound to the Fe3O4/MS system, as seen in Fig. 3b. The key to the sponge recovery is the adhesive, which is nano-Fe3O4. The PDMS@Fe3O4/MS surface in Fig. 3c is smooth, and the high water content of the sponge is due

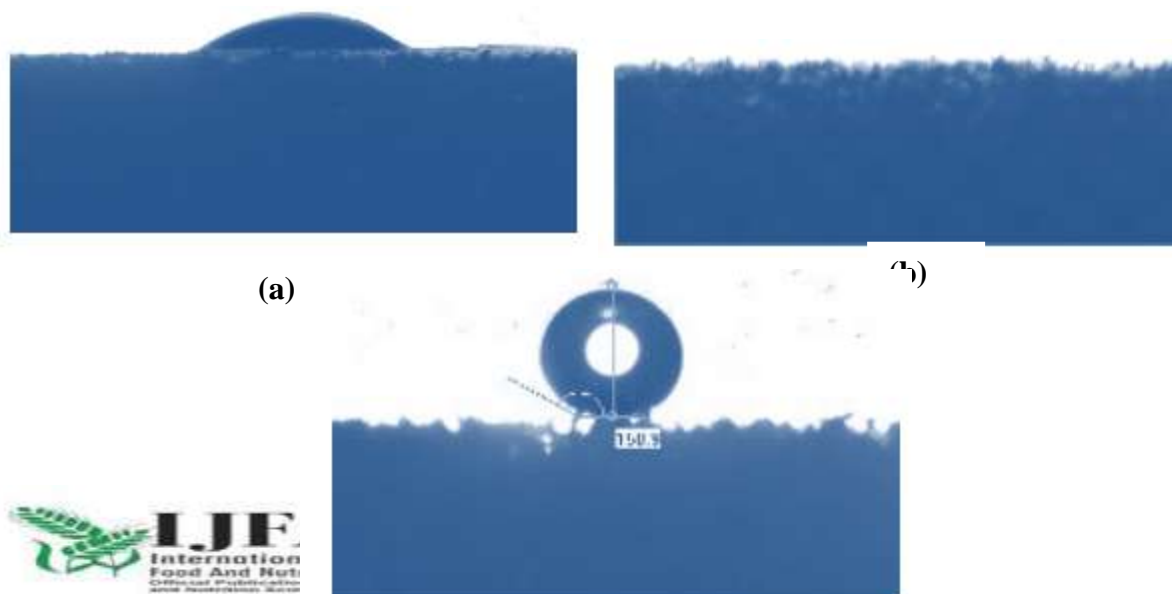
to the PDMS coating. This change dramatically increases the moisture content of the sponge. PDMS@Fe₃O₄/MS 40 μm, Fe₃O₄/MS, and MS scanning electron micrographs are shown in Figure 3d–f, respectively. All three figures have many open holes because they show a natural three-dimensional interaction. The MS has several natural pores that offer an effective system for oil-water separation, offering additional sites for the targeted application of oil.

Fig 2: MS, Fe₃O₄/MS, and PDMS@Fe₃O₄/MS scanning electron micrographs at 1 μm and 40 μm



The water contact with the melamine sponge before and after curing is displayed in Figure 3. Figure 3b displays the front view of the MS liquid contact surface. As soon as the water droplet contacts the sponge, it absorbs it because there is no contact angle, so the situation is superhydrophilic. A Fe₃O₄/MS/MSter contact diagram is presented in Figure 3a. The figure illustrates that the sponge does not absorb the droplets at all. This phenomenon indicates that, compared to MS, the water formation ability of Fe₃O₄/MS is better. However, this tensile strength is drastically reduced by the excess tensile strength. Figure 3c shows photographs of the water contacts formed by the modification treatment on the PDMS@Fe₃O₄/MS surface. The contact angle is 150.9°, and the droplets are circular on the sponge surface. The content has reached a superhydrophobic state if the water contact angle exceeds 150°. PDMS@Fe₃O₄/MS's potential for oil handling and oil-water separation is driven by its superhydrophobic environment.

Figure 3: Water contact angle images of Fe₃O₄/MS, MS, and PDMS@Fe₃O₄/MS are shown in (a), (b), and (c), respectively.



(c)

XRD experiments were conducted to visualize the crystal structure of nano-Fe₃O₄ and PDMS on the sponge. The outcomes are displayed in Figure 5 of the XRD experiments. The XRD pattern of PDMS@Fe₃O₄/MS indicates that the sponge skeleton is well coated by the PDMS coating. The unique diffraction peak in the PDMS crystal plane is located at 11.39° 2θ. The characteristic MS, appearing at a 2θ angle of 21.27° in the XRD pattern, corresponds to the standard JCPDS#24-1923 card. On the other hand, at 2θ of 21.27°, the Fe₃O₄/MS and PDMS@Fe₃O₄/MS XRD patterns are smooth. This phenomenon indicates that nano-Fe₃O₄ and PDMS hide distinct MS diffraction peaks. Typical nano-Fe₃O₄ diffraction peaks are located at about 35.14°, 55.78°, and 63.25°. Despite the fact that the specific MS diffraction peaks were unclear, there is ample evidence that nano-Fe₃O₄ is well integrated into the sponge skeleton. The unique diffraction peak of nano-Fe₃O₄ at 2θ 35.14° is the peak that has the standard card matching, JCPDF 0629, which proves its availability. This is supported by the absence of distinct diffraction peaks of MS at 35.14°, 55.78°, and 63.25°.

Oil-water separation test and altered sponge's capacity to absorb oil

Oil absorption in fresh sponge

We used formula (1) to determine the ability of PDMS@Fe₃O₄/MS to adsorb each of the three oil components. PDMS@Fe₃O₄/MS adsorption of corn oil is 17.99 0.26 g/g; 14.68 0.21 g/g for 0 # diesel; and 15.14 for kerosene is 0.38 g/g. Figure 7 shows the outcomes of the experiment. Table 1 shows the results of the experiment. The data in the table shows that, although PDMS@Fe₃O₄/MS and MS for 0# diesel have almost the same adsorption capacity, their initial difference is more than six times the same amount. This means its shrinkage will not decrease unless its core weight is greatly reduced. PDMS@Fe₃O₄/MS consequently has a high tensile strength.

Fig 4. The PDMS@Fe₃O₄/MS oil absorption capability for various oils

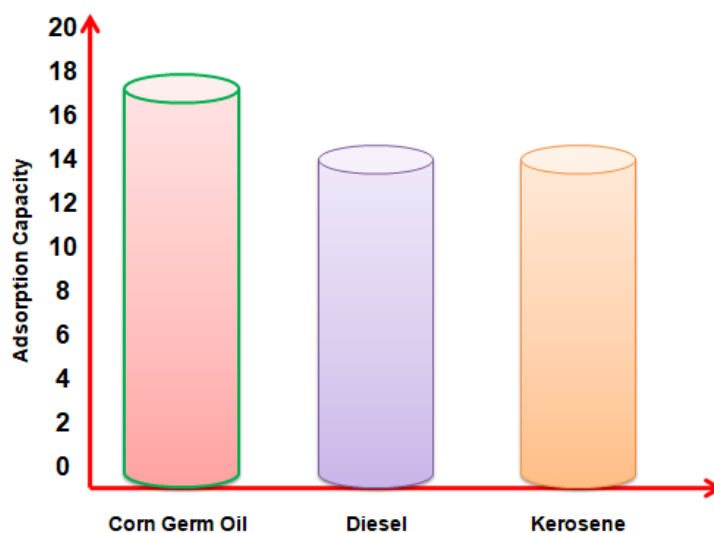


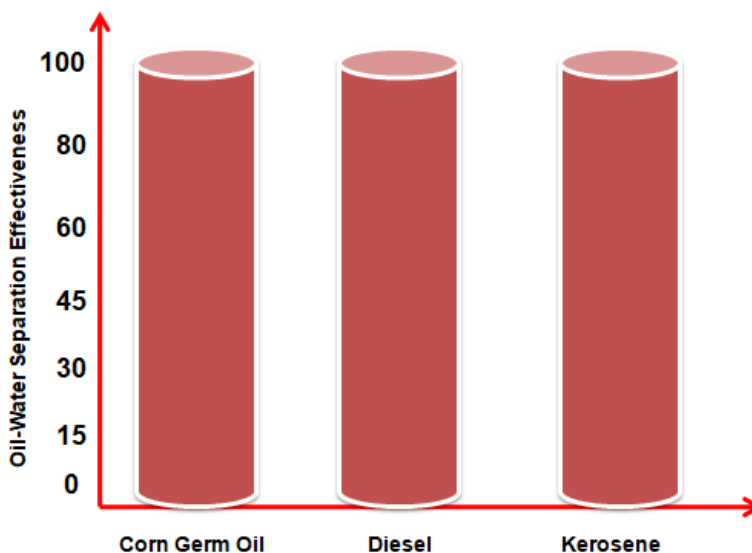
Table 1: Adsorption capacities of MS and PDMS@Fe3O4/MS are compared

Sponge	Initial Mass (g)	Mass after Adsorption (g)	Mass of Adsorbed Oil (g)	Adsorption Capacity (g/g)
MS	0.03	2.81	2.79	93.00
PDMS@Fe3O4/MS	0.19	2.94	2.75	14.49

Test for the modified sponge oil-water separation

Figure 5 displays the oil-water separation efficiencies of PDMS@Fe3O4/MS for blends of kerosene and water, diesel and water, and corn germ oil and water. 99.34 0.045%, 98.78 0.023%, and 98.80 0.019% The results were presented using formula (2). The high waterproofing properties of the PDMS coating and the three-dimensional porosity of the MS are responsible for its effective oil-water separation.

Fig 5: Effectiveness of PDMS@Fe3O4/MS separation for various oil-water blends

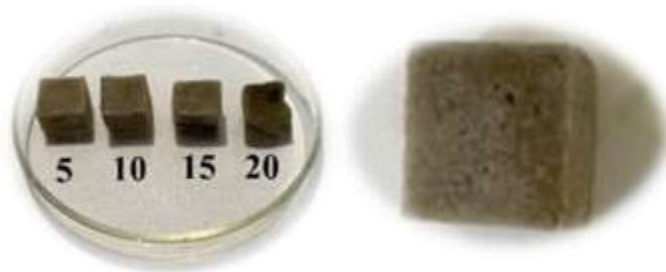


Performance test for oil-water separation and repeated oil absorption in the modified sponge

Mechanical squeezing of PDMS@Fe₃O₄/MS recycling was measured. After a number of predetermined adsorption processes, oil and water separation efficiency was determined. We demonstrated PDMS@Fe₃O₄/MS, which is an efficient oil-water separation and adsorption capacity at five-, ten-, and fifteen-fold oil extraction and after twenty. The potential for absorbing PDMS at Fe₃O₄/MS after five, ten, fifteen, and twenty oil-stripping cycles is shown in Table 2. The PDMS@Fe₃O₄/MS oil-water separation efficiency of five, ten, fifteen—it is the latter, as shown in Table 3—and twenty oil-breaking wheels. The graph in Figure 9 shows the variation of PDMS@Fe₃O₄/MS adsorption capacity during repeated oil sorption and oil stripping. The oil and water separation efficiency of PDMS@Fe₃O₄/MS is shown in Fig. 10 over time after repeated oil extraction and oil stripping.

PDMS@Fe₃O₄/MS experiences, after several cycles of oil absorption and deoiling, a decline in oil absorption capacity and oil and water efficiency of separation. This phenomenon has two primary causes. Initially, mechanical extraction disrupted the sponge density. The texture of the sponge has been greatly altered by repeated absorption and release of the oil. At the same time, the flexibility of the sponge begins to decrease. When mechanically pushed, it lost the ability to return to normal. The appearance of PDMS@Fe₃O₄/MS after five, ten, fifteen, and twenty deoiling cycles Second, the oil in the door cannot be fully emptied by mechanical squeezing. The sponge definitely includes some oil residue. The paper towel absorbs the remaining PDMS (Fe₃O₄)/MS oil.

**Fig 6: Apparition of PDMS@Fe₃O₄/MS after five, ten, fifteen, and twenty deoilings
& the remaining oil in the sponge is absorbed by the paper towel**



Experiment with a magnetically influenced modified sponge

A superhydrophobic melamine sponge floats on water when immersed in a beaker containing a specific volume of water. As the magnet slowly approaches the beaker, nano-Fe₃O₄ inside and on top of the sponge quickly moves towards the center and sides of the beaker. Due to this property, PDMS@Fe₃O₄/MS can be displaced depending on whether the adjacent oil is adsorbed or not. Once oil and water are separated in one area, the sponge is transported by magnetic force to additional regions containing oil. This sponge is able to remove the oil from

the water more effectively this way. The fact that it can be reused with the help of magnetism is also important. Sponges recovered after compression by the machine can be used, resulting in a significant decrease in the price of water and oil separation.

Conclusion

According to the melamine sponge, this paper describes the fabrication of a superhydrophobic magnetic melamine sponge through nano-Fe₃O₄ adsorption, PDMS modification, and oxidative polymerization of dopamine hydrochloride. Due to the PDMS layer, the sponge exhibits extraordinary superhydrophobicity. The findings indicate that the oil's contact angle is 0° and the water's contact angle is 150.9°. The sponge can absorb oily material fourteen times the volume of its own and can separate oil-water mixtures with filtering efficiencies of up to 99.34% in one session. It has a high absorption capacity for oil and water. In addition, the sponge may be recycled. The sponge was still able to absorb 12.57 times its oil after twenty cycles of oil absorption and deoiling, resulting in an efficiency of over 70% in the separation of oil from water. Also, the sponge can be magnetised with nano-Fe₃O₄. This improves the process of separating water and oil by letting the sponge move freely while being pulled by a magnetic field. However, workers may recycle the sponge using the same magnetic field. It is highly desirable to promote the widespread use of PDMS@Fe₃O₄/MS in waste oil production as well as recycling.

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