SANGOLA TALUKA SHETKARI SHIKSHAN PRASARAK MANDAL'S

Vidnyan Mahavidyalaya, Sangola

NAAC Re-Reaccreditation Grade “B”

Research publication and conferences activities-2019-20

In the academic year 2019-20 Dr. Mrs shinde published research article in moleculer symposia and presented research article at Fourth International conference on Advances in material sciences

Documents are attached as below

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Superhyrophobic PU Sponge modified by Hydrophobic Silica Nanoparticle – Polystyrene Nanocomposite for Oil-water Separation

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Keywords:	Superhydrophobic, nanocomposite, modified sponge, oil-water separation

Superhyrophobic PU Sponge modified by Hydrophobic Silica Nanoparticle – Polystyrene Nanocomposite for Oil-water Separation

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Abstract

In this study, we have synthesized the hydrophobic silica nanoparticles by simple sol-gel processing of polymethylhydrosiloxane (PMHS). The nanocomposite solution was prepared by adding hydrophobic silica nanoparticles in polystyrene (PS) solution and applied on the skeleton of polyurethane sponge by simple immersion – drying process. The as prepared sponges exhibited superhydrophobic property with water contact angle 161° and oil contact angle nearly 0° and could separate oil from oil-water and oil-muddy water mixture. The superhydrophobic sponge has sustainable anti-wetting property under cross sectional cutting, pressing and twisting, and different pH environment. Such superhydrophobic sponge is suitable for practical application on large scale.

Keywords: Superhydrophobic, nanocomposite, modified sponge and oil-water separation

Introduction

A spillage of oils in ocean and river results in a serious harm to the environment. In last century, oily waste water treatment technologies like air flotation [1], burning [2], gravity separation [3], membranes separation [4, 5] faced great challenges, such as secondary environmental pollution, low efficiency, time consuming and high cost. A new technology in materials science has been developed for easy oil-water separation using superhydrophobic nanomaterials. Like lotus leaf, on superhydrophobic surface, the water drop reveals the contact angle greater than 150° and roll off the surface quickly by little tilting. Recently, such superhydrophobic materials applied on mesh and sponges with special wettability are being utilized for efficient oil water separation process. The superhydrophobic and superoleophilic characteristic of the material illustrated to be used for oil water separation in many studies [6-11]. Due to extreme water repellent and quick oil absorbance property, this special wetting superhydrophobic materials shows the high separation efficiency and stability. However complex preparation process and high costs materials are restricting their industrial applications [12, 13].

Sponge is one of the low – cost three dimensional porous material with larger surface area and used as an ideal substrate for oil water separation. Wang et al [14] have applied hierarchical micro-nanostructured coating of polydopamine on melamine sponge by simple self-polymerization process and finally the sponge was modified by dodecanethiol to achieve superhydrophobic and superoleophilic wetting properties. The prepared sponges could easily separate oil layer from water as well as oil-water emulsion. The superhydrophobic sponges quickly absorbed various oils up to 20 times of its own weight. Xie and researchers [15] have prepared superhydrophobic melamine sponges by modifying with polydopamine and hydrophobic silica nanoparticles. These superhydrophobic sponges exhibited good mechanical durability, high adsorption capacity and excellent recyclability for more than 80 times. Moreover, the superhydrophobic sponges could continuously separate oil from salty water with ease. Gao et al [16] have used two step strategy like silica nanoparticle adsorption and silanization covering on melamine sponge to achieve water repellent properties. The sponges were durable against ultrasonication and corrosive environments and could effectively separate range of oils from water.

Zhang and researchers [17] have loaded hydrophobic thiolated grapheme on polyurethane sponge by simple dipping-drying process and such as prepared superhydrophobic sponges revealed high selectivity and recyclability. Cao et al [18] have used new carbon based material such as nanodiamonds to fabricate superhydrophobic sponges. The polyurethane sponge was coated by hydroxylated nanodiamonds with polydopamine and subsequent surface modification using 1H,1H,2H,2H-perfluorodecanethiol. These superhydrophobic sponges showed excellent oil-water separation property with high organic adsorption capacity. Li et al [19] have simply modified the polyurethane sponge by applying superhydrophobic attapulgite (APT) on the skeleton of the sponge. The prepared superhydrophobic sponges could separate oil from mixtures and various corrosive solutions and hot water. Beshkar et al [20] have modified the polyurethane sponge by simply immersing in the suspension of straw soot and magnetic nanoparticles. The as prepared sponges revealed superhydrophobicity and could separate various organic solvents from water with 30 times recyclability.

In this study, we have synthesized the hydrophobic silica nanoparticles by simple sol-gel processing of polymethylhydrosiloxane (PMHS). The nanocomposite solution was prepared by adding hydrophobic silica nanoparticles in polystyrene (PS) solution and applied on polyurethane sponge by simple immersion – drying process. The as prepared sponges exhibited superhydrophobic property and could separate oil from oil-water and oil-muddy water mixture.

Experiment Section

Materials

Poly(methylhydrosiloxane) (PMHS, Average Mn ~ 1,700 - 3,200) and polystyrene (PS, average Mw 192,000) were purchased from Sigma Aldrich. Ethanol (99.9% AR grade), chloroform (AR grade and sodium hydroxide pellets (AR grade) were purchased from Thomas Baker, PVT. LTD. India. Polyurethane (PU) sponge was bought from local market.

Sol-gel processing of PMHS

At first, 4.8 g PMHS was dissolved in 70 ml ethanol while stirring. A 0.08 g sodium hydroxide was dissolved in 2 ml distilled water and mixed in PMHS solution under constant stirring for 4 hour at room temperature. The sol was completely transformed into gel after 4 h. The gel was dried at 80°C for 5 h. The obtained powder was washed several times using ethanol and dried at room temperature for 24 h followed by grinding into a fine powder using mortar and pestle. The obtained hydrophobic silica nanoparticles were used to prepare the nanocomposite.

Preparation of Superhydrophobic Sponge

The PU sponges were washed repeatedly in ethanol and water for several times and dried at 80°C. The different nanocomposite solutions were prepared by dissolving 100 mg PS in 40 ml chloroform under constant stirring and added the 80, 120 and 180 mg of hydrophobic silica nanoparticles in PS solution separately under constant stirring. A PU sponge was immersed in the nanocomposite solution for 5 min and dried at 100 °C for 20 min and this procedure was repeated for 10 times. The sponges prepared with 80, 120 and 180 mg of hydrophobic silica nanoparticles concentration in nanocomposites are labeled as P1, P2 and P3 samples, respectively.

Characterizations

The microstructure of the sponge was stuydied by Scanning Electron Microscope (SEM, JEOL, JSM-7610F, Japan). The water and oil contact angles were measured using contact angle meter (HO-IAD-CAM-01, Holmarc Opto-Mechatronics Pvt. Ltd. India). To confirm the durability of the sponges, twisting, adhesive tape and sandpaper abrasion test were carried out. The oil-water separation ability of the sponges were analyzed by separating oil from oil-water mixture.

Results and Discussion

Morphology and Wettability of the Sponges

The microstructure of pristine PU sponge exhibits three dimensional interconnected micropores with diameter in the range of 150 - 500 µm (Fig. 1a) [21]. As the concentration of silica nanoparticles in the nanocomposite is very less in P1 sample, the separate aggregates of silica nanoparticles having sizes in the range of 1 to 10 µm were observed (Fig. 1b). However, the interconnected aggregates of silica nanoparticles were observed for P2 and P3 sample (Fig. 1c, d) due to higher concentration of silica nanoparticles in nanocomposite structure. The silica nanoparticles were bound together by PS. The P2 and P3 samples reveal rough surface morphology which is useful to achieve superhydrophobic wettability in the sponges.

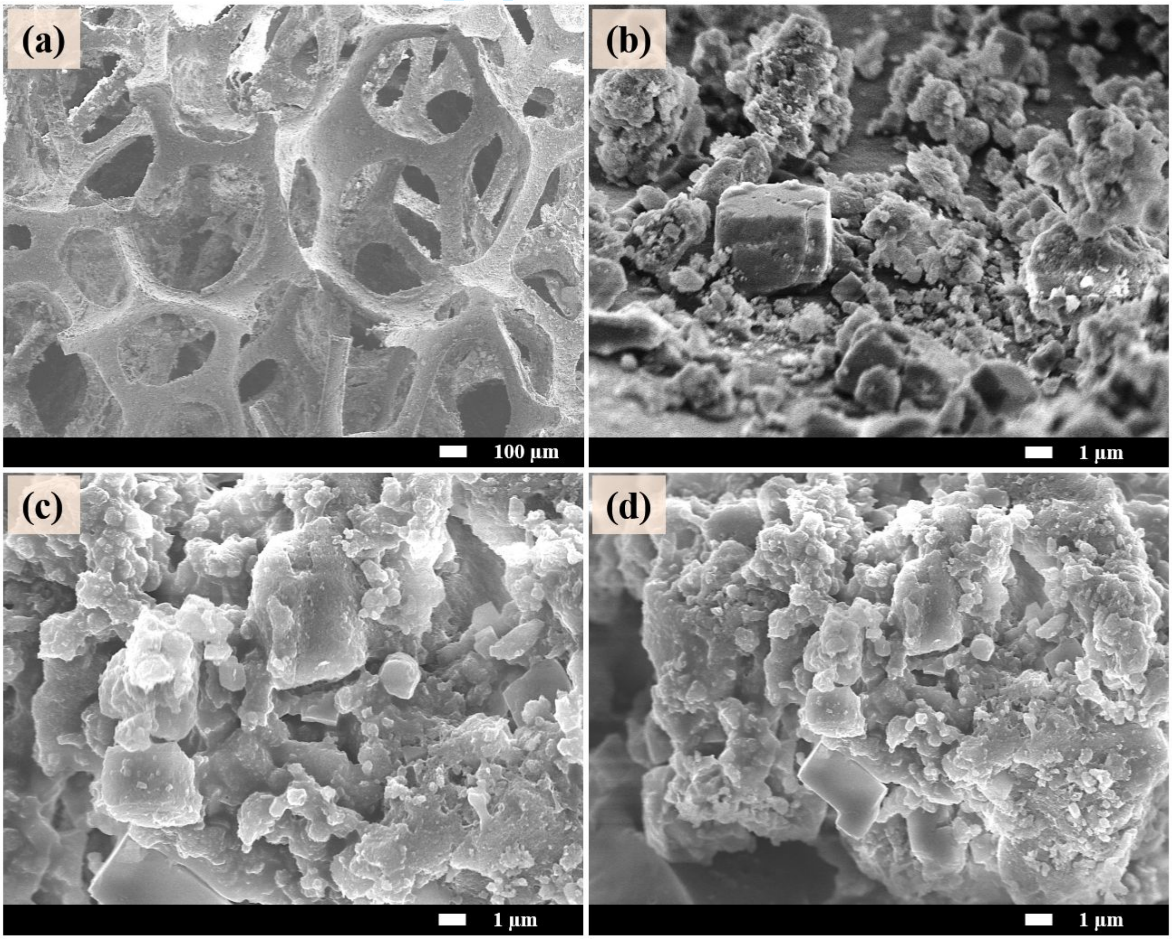

Fig. 1: SEM images of (a) pristine PU sponge, (b) P1, (c) P2, (d) P3 sample.

The wettability of the modified sponges was also depends upon the concentration of silica nanoparticles in the nanocomposite. The P1, P2 and P3 samples exhibited the water contact angle of 143°, 150° and 161°, respectively. Due to inhomogeneous coverage of nanocomposite structure on sponge, P1 sample show less water contact angle, whereas the nanocomposite material is uniformly and heavily got deposited on P2 and P3 samples resulting in water contact angle in superhdyrophobic state. All the samples exhibited superoleophilic property. Fig. 2 shows the optical photograph of water drops and oil (petrol) drops on P3 sample. An oil drop placed on the sample was quickly get absorbed inside the sponge with contact angle ~ 0° confirming its superoleophilic characteristic. Hence, P3 samples can be further used for efficient oil-water separation application.

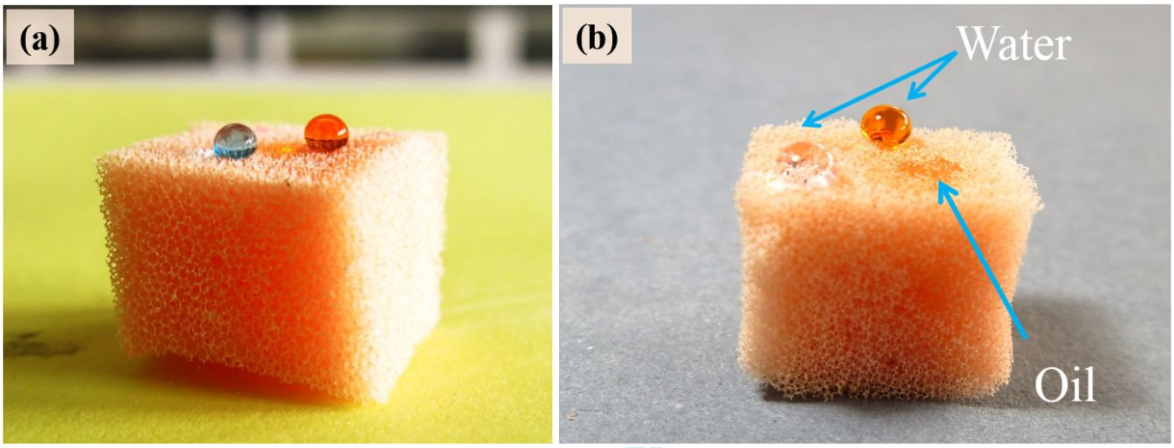

Fig. 2: Optical images of (a) water drops on P3 sample and (b) water and oil (petrol) drops on P3 sample.

Oil-water Separation Ability of Superhydrophobic Sponge

The absorption separation process of oil from oil-water mixture by superhydrophobic modified sponge is shown in Fig. 3. The oil-water mixture was prepared by adding 10 ml oil in 10 ml water. Three types of oil were used to study the oil-water separation. The superhydrophobic sponge was dipped in oil-water mixture and observed that superhydrophobic sponge quickly absorbed oil in few seconds. The oil absorbed spong pulled up and squeezed in another beaker to collect oil. Fig. 3 dipicts optical images of absorption and squeezing process of sponge. The oil-water seperation ability of sponge were tested by using disel (Fig. 3.a), kerosene (Fig. 3.b) and petrol (Fig. 3 c). So this promising, facile and energy saving method can be used to remove the oil from oil contaminated area.

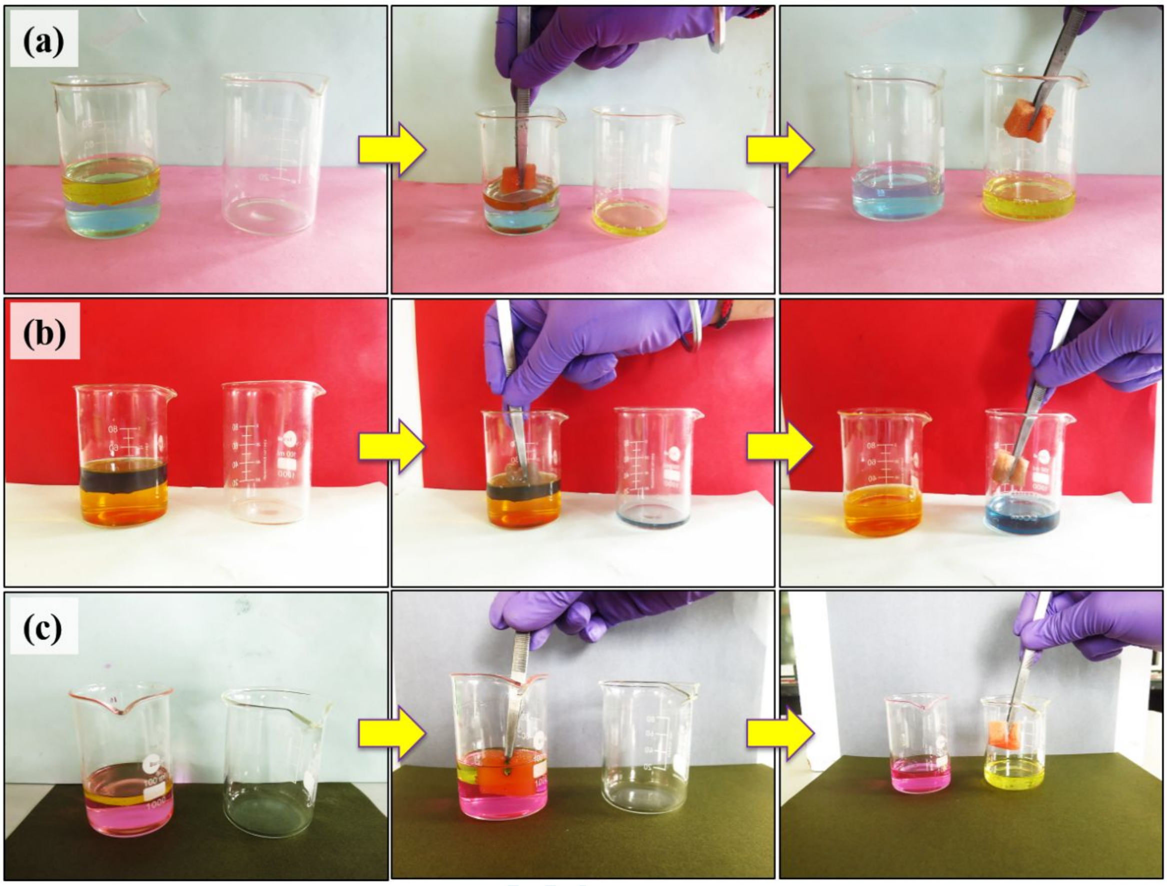

Fig. 3: Optical images of process of removal and collection of (a) diesel, (b) kerosene and (c) petrol from oil-water mixture.

In another way, the oil removal ability of sponge was tested using mixture of oil and muddy water. The muddy water was prepared by adding 10 gm soil in 50 ml water by stirring. A 5 ml kerosene was added in muddy water and stirred well to mix oil in it. After few second a layer of oil came on surface of muddy water. The oil was removed from muddy water by dipping superhydrophobic sponge in the oil-muddy water mixture (Fig. 4). The porous structure of superhydrophobic sponge able to absorb oil and repel muddy water completely.

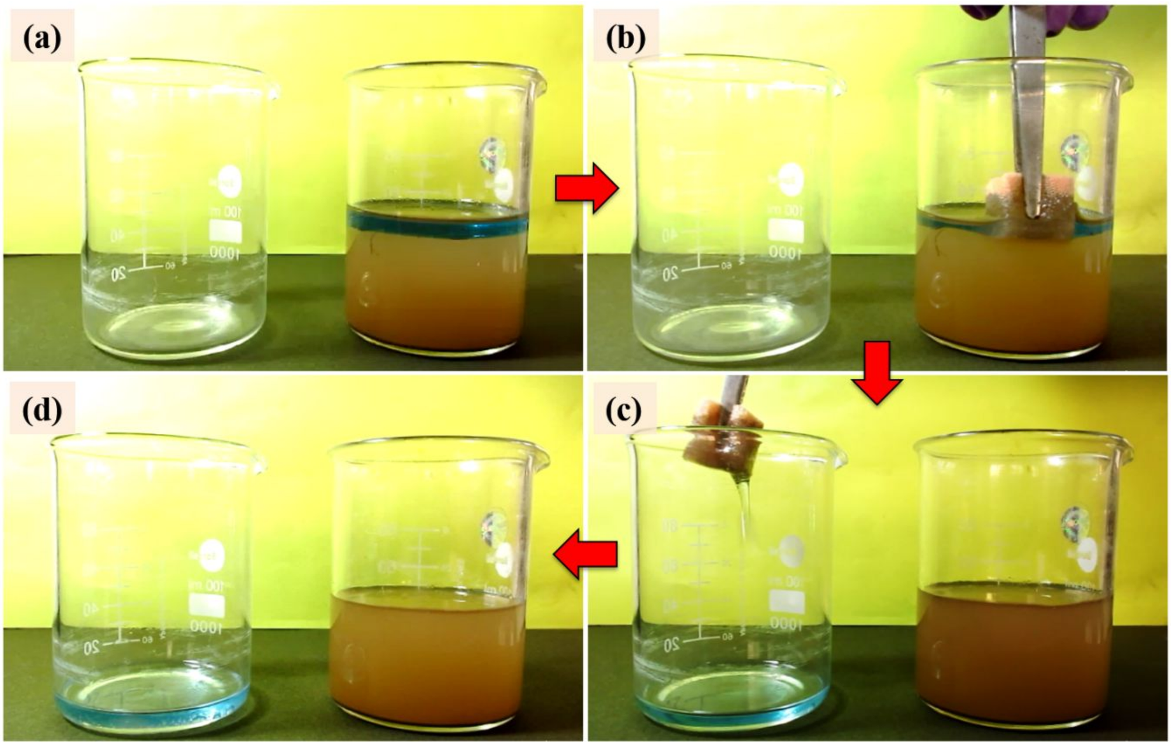

Fig. 4: Optical images of removal and collection of oil from the mixture of oil-muddy water by P3 sample.

Durability of anti-wetting property of superhydrophobic sponge

Several methods were adopted to investigate durability of anti-wetting property of superhydrophobic sponge such as adhesive tape and sandpaper abrasion test [22]. As shown in Fig. 5, the adhesive tape (Cellotape No. 405 having adhesiveness of 3.93N/10mm) was placed on P3 sample and metal disc of 200 gm was rolled on it to make good contact between the sponge surface and tape (Fig. 5 a and b). The tape was peeled off the sponge (Fig. 5c). After tape peeling test, sponge displayed superhydrophobic property with water contact angle 160° as shown in Fig. 5d. The sponge lost superhydrophobic property in 12 cycles of adhesive tape peeling test.

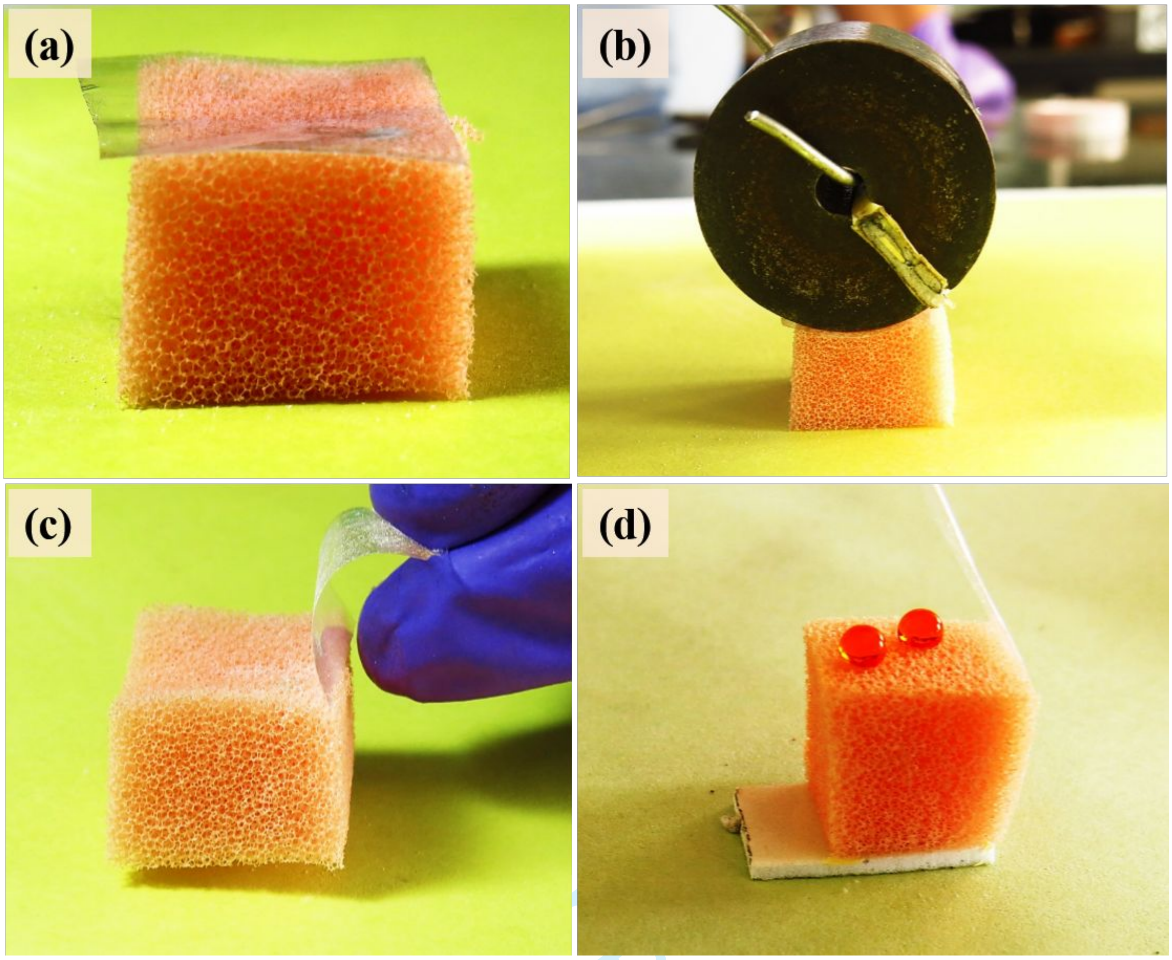

Fig. 5: Optical images of (a) Adhesive tape placed on (b) 200 g metalic disc rolling on tape. c) tape peeled off, and d) color water drop on P3 sample.

The abrasion resistance test was carried out by using sandpaper (Grit No. 320). The superhydrophobic P3 sample was placed on sandpaper with load of 100 g and dragged in linear speed for length of nearly 10 cm. This procedure is considered as one abrasion cycle and no contact angle change was observed on P3 sample. The sandpaper abrasion process is shown in Fig. 6 (a-d). After 20 cycles, the WCA on P3 sample was reached to 140° due to excessive damage to the surface by sandpaper. So it confirmed that the superhydrophobic sponge endure its wettability under adhesive tape and sandpaper abrasion test.

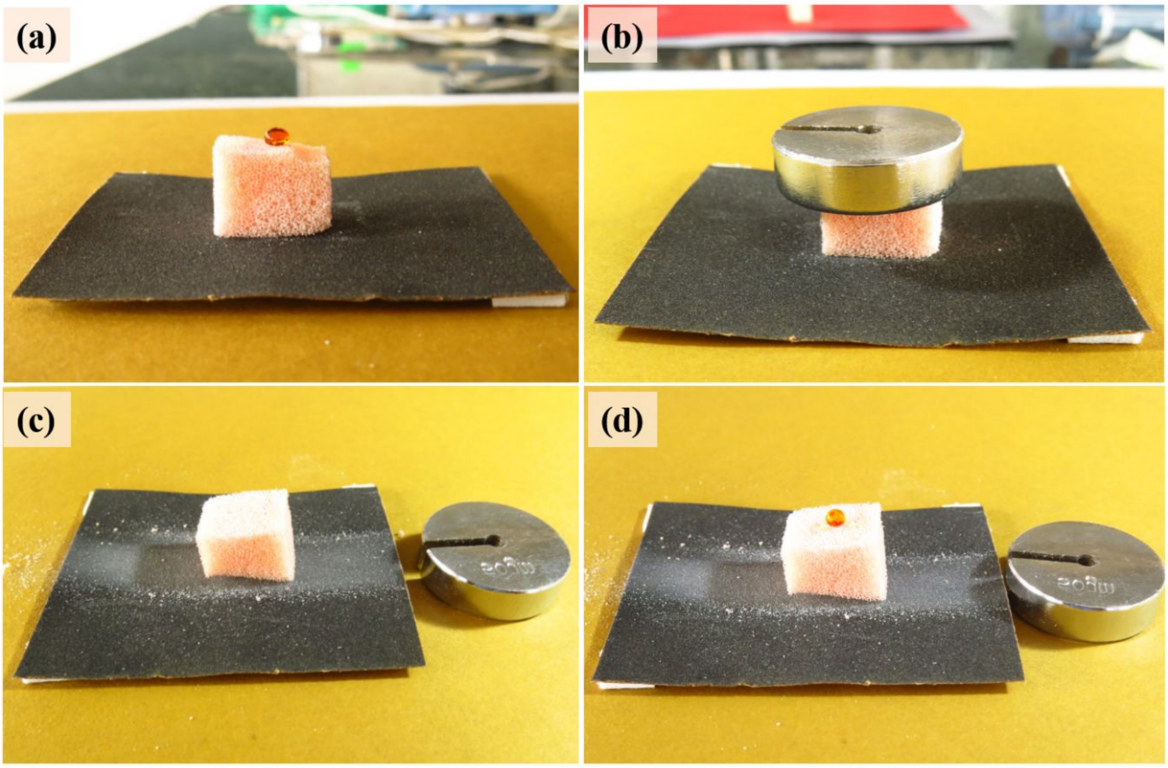

Fig. 6: Optical images of (a) P3 sample placed on sandpaper (b) 100g load applied on P3 sample, (c) abraded surface of P3 sample and (d) color water drop kept on abraded surface of P3 sample.

The wettability of superhydrophobic sponge was tested by taking cross section and twisting. The superhydrophobic sponge was cut in three different cross sectional positions, which look like stairs (Fig. 7 a). At every cross sectional face of sponge sustain its original surface superhydrophobicity, which showed simmilar contact angle as on initial surface of superhydrophobic sponge. The water drops on cross section of sponge as shown in Fig. 7 a. The superhydrophobic sponge pressed and twisted (Fig. 7b) and observed wettability. The water drop aquire spherical shape on affected area of sponge under twisting and pressing. Hence, the P3 sample sustain superhydrophobicity under mechanical insults.

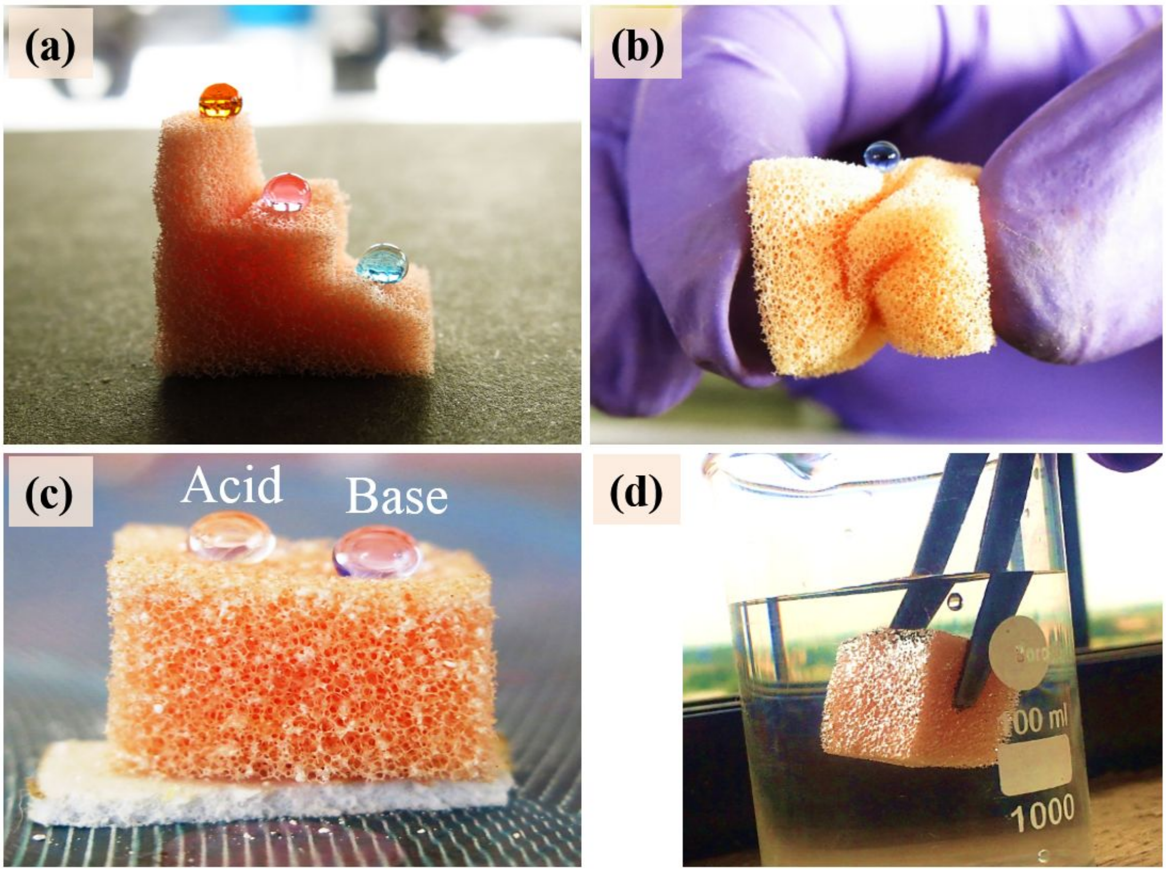

Fig. 7: Optical images of (a) water drops on cross section of P3 sample, (b) twisted sponge with color water drop on it, (c) acid and base drop on P3 sample, and (d) mirror like surface displayed when P3 sample immersed in water.

The chemical durability of superhydrophobic sponge was examined by different pH liquids. Acid and base drop (pH~3 and pH~11) showed spherical shape (Fig. 7c). Thus, superhydrophobic sponge successfully withstand in various chemical environment. When superhydrophobic sponge pushed in water, the mirror-like surface was observed (Fig. 7d). The bright surface due to total reflection of light from air layer present on surface of superhydrophobic sponge. The continues air layer indicates that Cassie-Baxter nonwetting behavior [23, 24].

Conclusion

In summary, sponge modified successfully by facile method using hydrophobic silica particles and polystyrene nanocomposite. The concentration of silica nanoparticles in nanocomposite affects on wettability of sponge. At optimum result sponge exhibit water contact angle 161° with superoleophilic (OCA~ 0°) property. The oil-water separation ability tested by three different oil including kerosene, petrol and diesel. Sponge showed good oil-water separation ability in all type of oil-water mixture and also good removal of oil from oil-muddy water mixture. Mechanical and chemical durability of modified sponge tested under cutting in cross section, pressing and twisting, and different pH. In the durability test modified sponge has excellent anti-wetting property. This superhydrophobic sponge suitable for large-scale oil/water separation in practical applications, such as, water purification and environmental oil spills.

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Fourth International Conference on Advances in Materials Science

(ICAMS - 2020) on 20 – 21 January 2020

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This is to certify that Prof. Dr. Shinde Shital Rajaram of Vidnyan Mahavidyalaya Sangola has worked as an **Examiner** for **Poster Session** at the **Fourth International Conference on Advances in Materials Science (ICAMS – 2020) on 20 - 21 January 2020** organized by Post – Graduate Department of Physics, Raje Ramrao Mahavidyalaya, Jath – 416 404, Dist – Sangli, Maharashtra, India.

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This is to certify that ~~Mr.~~/Miss./Dr. **Shital Rajaram Shinde** has participated and presented a paper entitled "**Superhydrophobic PU Sponge Modified by Hydrophobic Silica nano particle-Polystyrene Nanocomposite For Oil Water Separation**" in ***~~Oral~~/Poster Session*** of the Fourth International Conference on Advances in Materials Science (ICAMS – 2020) on 20 - 21 January 2020 organized by Post – Graduate Department of Physics, Raje Ramrao Mahavidyalaya, Jath – 416 404, Dist – Sangli, Maharashtra, India.

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