

# Superhydrophobic Sponge Modified By Naturally Synthesized Silica And Candle Soot Nanoparticles

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**Abstract-** *Economic and eco-friendly superhydrophobic sponges were readily fabricated by coating a candle soot-wax composite for efficient oil-water separation. For this, the candle soot was extracted from the combustion of a paraffin candle and deposited onto sponges using wax by a dip-coating process. This led to the adhesion of candle soot and producing competent superhydrophobic sponges. It exhibits a water contact angle of  $>150^\circ$  that informs remarkable superhydrophobicity of the surface. Its superiority was tested with several parameters including thermal stability, pH tolerance, compression tolerance, chemical durability, reusability, emulsion oil-water separation, and muddy water-oil separation offers satisfactory results besides validating its superhydrophobic nature. Furthermore, its superhydrophobic tolerance was analyzed by testing for anti-wetting properties, cross-sectional cutting, pressing, paper peel test, and abrasion resistance. So, this simple candle soot-wax coating technology has practical capabilities for coating large surfaces. This promising material can work as a distinct and economical oil-water separator, which will help to prevent massive environmental pollution caused by oil spills in water.*

**Keywords-** Superhydrophobic, Candle Soot, Oil-Water Separation, Contact Angle.

## I. INTRODUCTION

The separation of oil and water has a big problem in the world. The huge amount of water is polluted due to blending of oil with water, which produced in daily life and industrial field. The many sources of oily-water such as oil spill accidents, marine environment, aquatic ecosystem, wastewater in industrial process such as petroleum, daily chemical, textile, leather and steel processing and metal finishing and so on.

The oily wastewater is defined by the quantity of oil in water and they are named as floated oil and dispersed oil (about 90 wt %), emulsified oil (about 10 wt %), and dissolved

oil (<0.5 wt %) [1- 2]. The floated oil and dispersed oil easily separate oil and water mechanical process, but very difficult to separate emulsified oil and dissolved oil because this has an oil droplet size  $< 10 \mu\text{m}$  [2, 3]. There are some traditional ways for the separation of oil and water including gravity separation and skimming, air-flotation, coagulation, de-emulsification, and flocculation [4-6]. These techniques have intrinsic disadvantages such as low efficiency, high operation cost, corrosion, and re-contamination problems. On other some absorbent materials like zeolites [7], activated carbon, hair [8], organoclays [4], gelators derived from sugar [9] or straw [10], carbon nanotube sponges [11], wool fibers [12], and others [13] have been used as an oil–water separator.

The wettability of solid materials has been controlled by superhydrophobic functional materials using a combination of low surface energy and rough topography. The superhydrophobic property is usually characterized by a liquid contact angle above  $150^\circ$  and a sliding angle below  $5^\circ$ . In nature, the lotus leaf is a well-known example of superhydrophobic and self-cleaning. Wenzel [14] and Cassie-Baxter explain the structure of superhydrophobic surfaces. In superhydrophobic surfaces, air pockets are trapped inside the rough surface, which minimizes liquid and solid contact area. So, the liquid droplets were easily movable on a superhydrophobic surface. There are a lot of essential applications of superhydrophobic surfaces including self-cleaning windshields, prevention of snow adhesion, anti-biofouling, anti-fogging, and antifouling architectural coatings [15]. The superhydrophobic materials (sponge and membrane) play an important role in oil-water separation.

Superoleophobic is one of the important wetting properties of solid materials. The superhydrophilic/superoleophobic sponges and membranes could separate oil and water. In recent years, the superhydrophobic and superoleophobic porous materials have attracted fundamental research and application in oil and water separation. Metal meshes, textiles, sponges/foams, and porous

polymers play important roles in porous superhydrophobic and superoleophobic materials. The water floats on the surface of superhydrophobic material but quickly absorbs or goes through the membrane due to superoleophilic properties, so it spontaneously separates oil and water.

## II. EXPERIMENTAL WORK

### Material:

Commercial polyurethane sponges, coconut husk, and candles were purchased from a local supermarket. H<sub>2</sub>SO<sub>4</sub>, NaOH, and HCl were purchased from Shri Samarth Commercialism Company, Pvt. Ltd., India. Organic solvents including toluene, hexane, and chloroform were purchased from Shri Samarth Commercialism Company, Pvt. Ltd., India. All the chemicals used for the experiments were standard commercial grade, which were used as received without any further purification.

### Method:

#### 1. Preparation of hydrophobic SiO<sub>2</sub> nanoparticles:



**Coconut Husk**



**Coconut Husk Ash**

#### Acid washing:

- An acid washing step was used to remove impurities from waste products to silica extraction.
- 10 g of coconut husk dispersed in water and then washed out 10 % HCl.
- Then these waste products are dried on a hot plate for half an hour.
- The dried husk was subjected to heat treatment in a muffle furnace at 600°C for an hour to obtain ash.

#### Extraction of silica:

- In the sample of 10 g ash, add 1N NaOH solution to it and stir for two hours at 90°C constantly.
- These solutions were filtered through Whatman filter paper and the obtained viscous and transparent

filtrated solution was allowed to cool at room temperature.

- Prepared 1N HCl solution was added to the extracted solution from the burette with dropwise addition under constant stirring till it solved coconut husk ash.



- The coconut ash filtrate, the prepared 2.5 N H<sub>2</sub>SO<sub>4</sub> solution adjust to burette, under constant stirring adding a dropwise H<sub>2</sub>SO<sub>4</sub> to the filtrate until the pH is neutral.
- Again, these neutral filtrates are stirred on a magnetic stirrer for two hours at 90°C to obtain a white Gelish precipitate.
- When the precipitate is settled down these filtrates were allowed to hot plate about 100°C. All filtrate was vaporized and the whitish dried silica was obtained in powder form.
- This silica powder was collected in dried different three bottles.



**Silica Gel**

**Silica Powder**

**Silica Powder**

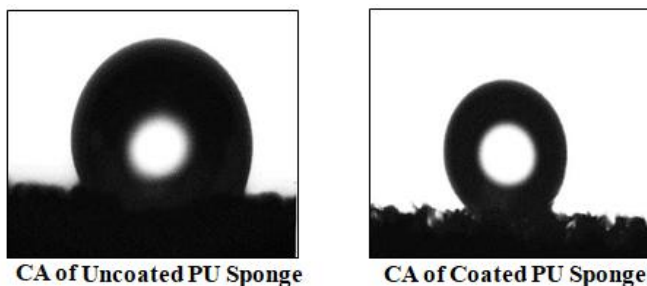
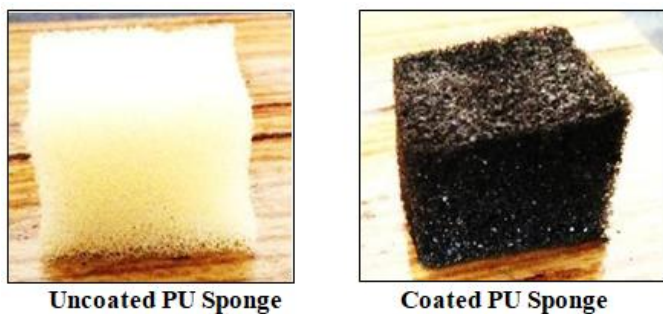
**2. Collection of superhydrophobic CS:**

The growth of CS was performed using a combustion flame process of paraffin candles in open air. When incomplete combustion was deliberately induced by blocking the flame of a candle, hydrophobic CS was generated as a by-product. CS was collected by placing a beaker on top of the flame of a paraffin candle mid-flame position. Subsequently, the collected CS was transferred into the crucible. The CS collected from the mid-flame of burning candles has remarkable superhydrophobicity and good performance in repelling hot water. The reason is that the centred flame is not sufficient combustion and organic compounds are enriched with carbonaceous materials.



**3. Preparation of superhydrophobic/superoleophobic CS-SiO2-PU sponge:**

The CS-SiO2-PU sponge was prepared by a facile dip coating method. The sponges of 3 × 2 × 1 cm<sup>3</sup> were dipped in deionized water, acetone, and ethanol, respectively by ultrasonic wave washing at room temperature. Subsequently, the pre-cleaned sponge was immersed in a dispersion of SiO<sub>2</sub> and CS in toluene and after magnetic stirring for 2 h, was dried in the oven at 60°C for 2 h. The relationship between the mass ratio of SiO<sub>2</sub>/CS NPs and the water CAs on the as-prepared sponge was measured. We can gain the different SiO<sub>2</sub>/CS NPs loading by changing the dispersion amount.



**III. RESULT AND DISCUSSION**

**1. Water Contact Angle:**

The relationship between the mass ratio of SiO<sub>2</sub>/CS NPs and the water CAs on the as-prepared sponge was measured which is shown in the table below:

**Table 1:** The relationship between the mass of SiO<sub>2</sub>/CS NPs and the CAs of water on the CS-SiO<sub>2</sub>-PU sponge.

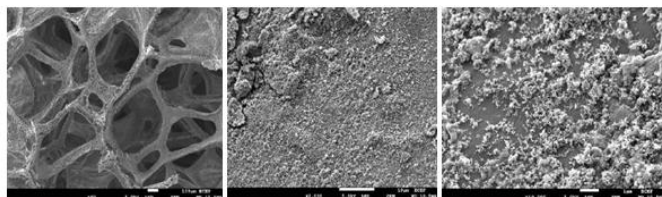
SiO <sub>2</sub> (g)	Candle Soot (g)	SiO <sub>2</sub> :CS	Water contact angle (°)
0.020	0.005	4:1	130.2
0.020	0.010	2:1	136.8
0.050	0.010	5:1	140.9
0.050	0.020	5:2	143.6
0.050	0.030	5:3	142.4
0.100	0.010	10:1	147.5
0.100	0.020	10:2	146.6
0.100	0.030	10:3	148.8
0.150	0.010	15:1	152.2
0.150	0.020	15:2	155.9
0.150	0.030	15:3	154.7
0.200	0.010	20:1	155.8
0.200	0.020	20:2	155.6
0.200	0.030	20:3	155.2

**FTIR:**

The peaks at 2920 and 2848  $\text{cm}^{-1}$  correspond to the asymmetric and symmetric stretching vibration of  $\text{CH}_2$  groups, respectively. The peaks at 1000–1300  $\text{cm}^{-1}$  were assigned to the Si–O–Si stretching vibration. These indicated that a stable monolayer of CS has already come into being on the surface of the  $\text{SiO}_2$  nanoparticles.

**SEM:**

The surface morphology of the PU sponge and the as-prepared sponges is investigated by SEM at different magnifications, as shown in Fig. It is observed that the PU sponge has a three-dimensional hierarchical porous structure with pore sizes ranging from 100 to 300  $\mu\text{m}$  and the high magnification of the image in the inset of exhibits a smooth surface of sponge skeletons, shows the SEM images of the CS- $\text{SiO}_2$ -PU sponge at low and high magnifications. Numerous microscale aggregates are covered on the skeletons of the sponge, indicating that CS and  $\text{SiO}_2$  NPs have been coated on the sponge successfully. The higher magnification reveals that lots of CS and  $\text{SiO}_2$  NPs aggregate and form micro- nano-rough structures on the skeletons of sponges. The 3D micro-porous structure of the sponge and the nanoscale CS and  $\text{SiO}_2$  NPs form a binary rough structure, which is extremely similar to the structure of lotus leaves.



**Fig. 3.** SEM images of an initial PU sponge. The inset is the high-magnification image of the PU sponge and the CS- $\text{SiO}_2$ -PU sponge surface morphologies at low and high magnifications, respectively.

**IV. CONCLUSION**

In conclusion, we successfully prepared a stable superhydrophobic sponge by a facile solution dip coating method for clean-up of oil contamination. The CS- $\text{SiO}_2$ -PU sponge possesses stable superhydrophobicity and excellent ability of selective absorption to oil even at various harsh conditions, including acid, alkali, and salt aqueous solutions at mechanical agitation conditions, hot water, and ice/water mixtures. Finally, the CS- $\text{SiO}_2$ -PU sponge, combined with a vacuum system, could continuously absorb and remove oil from the water's surface. The CS- $\text{SiO}_2$ -PU sponge possessed remarkable performance, including a facile fabrication method, high separation efficiency, good recyclability, anti-

corrosion, and excellent superhydrophobicity for hot water, which demonstrated that CS- $\text{SiO}_2$ -PU sponge as an absorptive material has significant value in water remediation for practical applications.

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