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Embellishing 2-D MoS₂ Nanosheets on Lotus Thread Devices for Enhanced Hydrophobicity and Antimicrobial Activity

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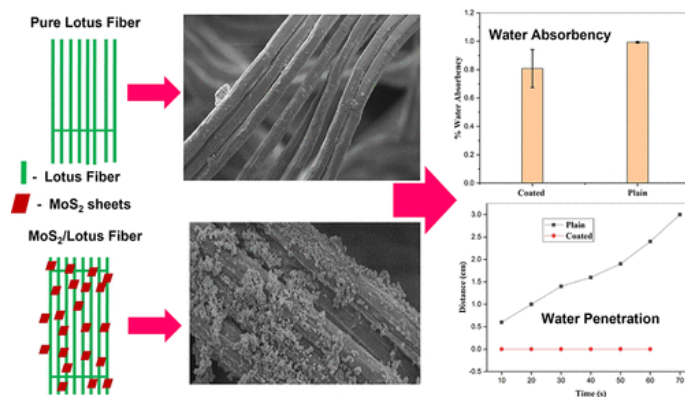
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SUBJECTS: Absorption, Cellulose, Fibers, Hydrophobicity, Two dimensional materials

Abstract



Herein, we report cellulose-based threads from Indian sacred Lotus (*Nelumbo nucifera*) of the Nymphaeaceae family embellished with MoS₂ nanosheets for its enhanced hydrophobic and antimicrobial properties. MoS₂ nanosheets

X-ray diffractometry (XRD) and scanning electron microscopy (SEM). The XRD pattern of pure lotus threads showed a semicrystalline nature, and the threads@MoS₂ composite showed more crystallinity than the pure threads. SEM depicts that pure lotus threads possess a smooth surface, and the MoS₂ nanosheets growth can be easily identified on the threads@MoS₂. Further, the presence of MoS₂ nanosheets on threads was confirmed with EDX elemental analysis. Antimicrobial studies with *Escherichia coli* and *Candida albicans* reveal that threads@MoS₂ have better resistance than its counterpart, i.e., pure threads. MoS₂ sheets play a predominant role in restricting the wicking capability of the pure threads due to their enhanced hydrophobic property. The water absorbency assay denotes the absorption rate of threads@MoS₂ to 80%, and threads@MoS₂ shows no penetration for the observed 60 min, thus confirming its wicking restriction. The contact angle for threads@MoS₂ is 128°, indicating its improved hydrophobicity.

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1. Introduction

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Lotus, an aquatic perennial widely cultivated in India, Asia, Australia, China, and Japan, typically grows in swamps and shallow waters. (1) The natural cellulose from lotus fibers is associated with continuous rings inside the peduncles lying under the epidermis of vascular tissue. (2–5) The lotus leaf's excellent and stable superhydrophobicity is due to a combination of optimal traits such as surface topography, toughness, and the epicuticular wax's unique qualities. The Lotus effect has encouraged researchers to create superhydrophobic surfaces and to design materials with enhanced hydrophobicity obtained from the lotus fibers. (6)

Nanomaterials have played a prominent role in altering size and structure at the nanoscale level to achieve and mimic the property mentioned above. 2D materials are currently recognized as nanomaterials having a sheetlike shape and a substantial lateral dimension ranging from hundreds of nanometers to tens of micrometers or even greater but only a single or few atomic layer thickness. Transition-metal dichalcogenides, noble metal dichalcogenides, MXenes, hexanol boron nitride, organics/polymers, and transition-metal halides are some examples of innovative 2D materials beyond graphene. (7) Of these, transition-metal dichalcogenides (TMDCs) have a one-of-a-kind amalgamation in-direct bandgap, approving electronic and mechanical properties, spin-orbit solid coupling, and thickness on the atomic scale, making them appealing for elementary research as well as applications that include personalized medicine, flexible electronics, high-end electronics, energy harvesting, DNA sequencing, optoelectronics, and spintronics. (8,9) TMDCs are made up of three atomic planes and often two atomic species: a metal and two chalcogens. TMDCs have a generic formula of MX₂ where M denotes transition metal and (M = V, Zr, Ti, Ta, Hf, Nb, W, Co, Tc, Ir, Re, Pd, Rh, Ni, Mo, and Pt) and X denotes chalcogen (X = Te, S, and Se). The layered metal chalcogenides encompass a wide range of electrical characteristics from real metals (NbS₂) to superconductors (TaS₂) to semiconductors (MoS₂) with a wide variety of bandgaps and offsets. (9,10)

Abstract

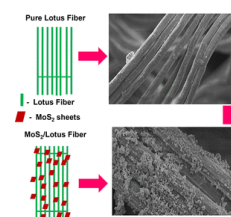


Figure 1

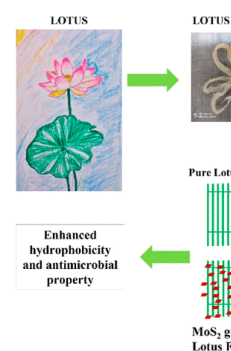


Figure 1. Schematic illustration of the process of coating 2D-MoS₂ nanosheets onto lotus threads.

Figure 2

(11–13). Since then, several nanoscience and nanotechnology journals have focused on the area of 2D materials. MoS₂ possesses a hexagonal arrangement consisting of S–Mo–S covalent bonds, and between the neighboring layers of MoS₂ there is a van der Waals interaction that allows them to be mechanically separated to form two-dimensional nanosheets. (14). The two-dimensional MoS₂ nanosheets have various physical and chemical properties and possess several applications. Recent research on MoS₂ has revealed this as a solitary contender in hydrogen storage, supercapacitors, sensors, electrocatalysis, and other applications such as electronic sensors, biomedical engineering, and other applications. The remarkable unique properties include a great amount of surface area and absorption in the near-infrared band, thus providing a new outcome in biological applications. (15). Biomedical uses for 2D MoS₂ sheets have been recently explored as well. In their seminal work, Zhu et al. explained that MoS₂ monolayers could be used to identify DNA molecules based on their fluorescence quenching capabilities. MoS₂ sheets have been employed as an NIR photothermal agent to kill Hela cells using their near-infrared (NIR) absorption. It has been reported that PEG-functionalized MoS₂ sheets can be used to transport drugs. (16).

The utilization and manipulation of the thread's wicking qualities for building programmable microfluidic channels have been the focus of thread-based research. So far, researchers have been looking for appealing substrate materials for decades to keep microfluidics advancing and overcome the disadvantages and difficulties such as tedious and expensive fabrication methods. Because of their unique structural and mechanical qualities, cellulose substrates such as thread and paper are considered as viable solutions for various applications. (17–21). Thread has demonstrated many potential applications in diagnostic systems, smart bandages, and tissue engineering. (22). Thread-based microfluidics is still in its infancy, and additional developments in manufacturing, analytical methodologies, and function are required before they can be commercialized as low-cost, low-volume, and simple-to-use point-of-care (POC) diagnostic devices. (23–26). Because of its features like flexibility, portability, biodegradability, lightweight, high tensile strength, and availability, several attempts have been made to employ thread for low-cost diagnostics or detection, among other low-cost materials such as paper and plastic. (27–30). Liquid wicking in the thread is caused by the twisted strands of cellulose fiber and the space between them.

In this work, for the first time, we have incorporated 2D TMDC MoS₂ nanomaterials on natural threads obtained from lotus fibers (Figure 1). Since MoS₂ nanocomposites are widely used for diode fabrication, (31) dye removal processes, (32) high-performance microwave absorbers, (33) fuel oil separation, (34) tunable microwave absorbers, (35) and electromagnetic wave absorption capability, (36) the idea of drop-casting 2D-nanomaterials on a cellulose fiber can offer a different perspective for wearable sensors. Integration of thread devices (natural and synthetic) with 2D nanomaterials for enhanced hydrophobicity and antimicrobial activity remains unexplored. There has been an increasing interest in discovering and producing novel antimicrobial agents from numerous sources in recent years to tackle microbial resistance. As a result, antimicrobial activity screening and evaluation methodologies have received more attention. (37). Antimicrobial susceptibility

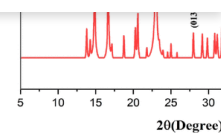


Figure 2. Comparative of lotus fiber and cellulose β cellulose patterns.

Figure 3

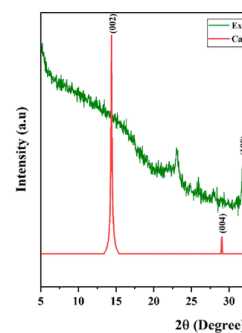


Figure 3. Comparative of MoS₂@fiber and cellulose β cellulose MoS₂.

Figure 4

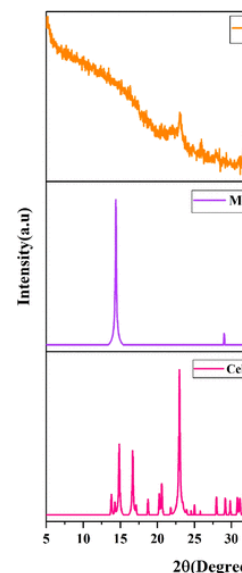


Figure 4. Comparative of fiber@MoS₂ with CIF P 4114994 and 9007660 and MoS₂, respectively.

Figure 5

threads@moS₂ are assessed for their potential antimicrobial properties against *Escherichia coli* and *Candida albicans* under light and dark conditions. MoS₂ was synthesized using the coprecipitation technique and further characterized through XRD and FESEM. (39).

Figure 1

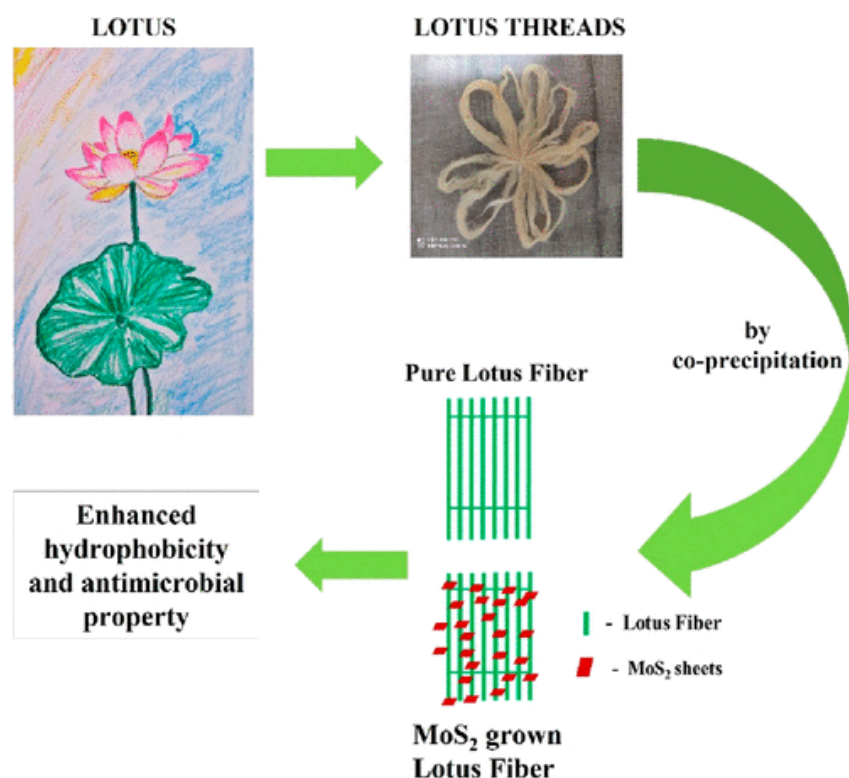


Figure 1. Schematic illustration of coating 2D-MoS₂ nanosheets on lotus threads.

2. Experimental Methods

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2.1. Materials Used

Chemicals used in this research work were used as purchased. Sodium molybdate dihydrate (Na₂MoO₄·2H₂O, Sisco laboratories, 99%), thioacetamide (CH₃CSNH₂, Loba Chemie, 99%), and hydrochloric acid (HCl, Merck Life, 37%) were purchased. Standard strains of *E. coli* (ATCC 25922) and *C. albicans* (ATCC 24433) were obtained for testing antimicrobial properties from the Department of Microbiology, Kasturba Medical College, Manipal. Nutrient Agar and Sabouraud Dextrose Agar with chloramphenicol were procured from Himedia, India.

2.2. Extraction of Lotus Fiber

Lotus stems were collected at Kolarampathy Lake in Coimbatore, Tamil Nadu, with a latitude of ~10.973400° and longitude of ~76.909850°. Ideally, flowers should be fully bloomed so that the deep pink blooms contain the finest lotus fibers. The collected fibers are then trimmed, snapped, and twisted. The twisted fibers reveal 20–30 fine white filaments pulled and wrapped into a single thread.

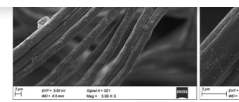


Figure 5. Different magnification SEM images (a–d) of lotus fiber.

Figure 6

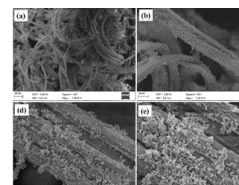


Figure 6. Different magnification SEM images (a–e) and surface morphology of MoS₂-coated lotus fiber.

Figure 7

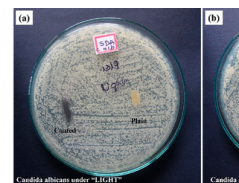


Figure 7. Antifungal activity of pure and MoS₂-coated lotus fiber under light (a) and dark (b) conditions.

Figure 8

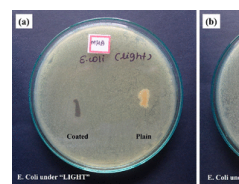
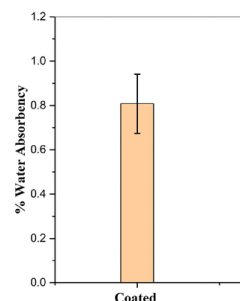


Figure 8. Antibacterial activity of pure and MoS₂-coated lotus fiber under light (a) and dark (b) conditions.

Figure 9



mmol of CH_3CSNH_2 was added to the above solution. The well-washed (with Millipore water and ethanol) lotus fiber thread was dipped inside the solution, and then the solution was heated to $65\text{ }^\circ\text{C}$. HCl was included dropwise to the mother solution at $65\text{ }^\circ\text{C}$. The colorless solution turned dark blue. The heat treatment continued, and a color change from dark blue to brown and then eventually to chocolate brown within 10 min of adding HCl was observed. The temperature of the solution was maintained at $80\text{ }^\circ\text{C}$ for 1 h. The particles were left overnight for the settlement. The collected particles, which were then centrifuged for 10 min at 3500 rpm, were washed and dried at $55\text{ }^\circ\text{C}$ and collected.

2.4. Characterization

The structure of the coated fiber was examined by a Philips PAN analytical Xpert pro powder X-ray diffractometer with $\text{Cu K}\alpha$ (1.54 \AA). Morphology and elemental analysis of pure and MoS_2 -coated lotus fiber were recorded using an S-3400 N Hitachi field emission scanning electron microscope (FESEM).

The hydrophobicity of the uncoated and MoS_2 nanoparticle-coated lotus fiber threads (3 cm length) was assessed by measuring the water penetration rate in the thread pieces. A $100\text{ }\mu\text{L}$ portion of phenol red dye solution in water was added to one end of the threads placed over an overhead projector (OHP) sheet, and images of the threads were captured at defined time intervals using a Canon Eos 3000D DSLR camera and further analyzed using FIJI software.

The water absorbency of the uncoated and coated fibers (1 cm length) was determined by measuring the dry weight of the threads using a weighing balance then dipping the thread pieces in 1 mL of water for 5 min to measure the wet weight of the threads. The percentage of water absorbency was measured using the following formula:

$$\% \text{ water absorbency} = \frac{\text{wet weight} - \text{dry weight}}{\text{wet weight}} \times 100 \quad (1)$$

The contact angle measurements for the uncoated and coated fibers were analyzed using the KYOWA Interface Measurement and Analysis System through a sessile drop method.

2.5. Antimicrobial Properties

Culture suspensions of *E. coli* and *C. albicans* spiked in water were prepared, adjusted to 0.5 McFarland standard concentration, and inoculated on Muller Hinton Agar (MHA) and Sabouraud Dextrose Agar (SDA) with chloramphenicol, respectively. The uncoated and MoS_2 nanoparticle-coated lotus fiber threads (UV sterilized, 10 mm length) were placed on the agar media in the inoculated plates and incubated under two different conditions to check the antimicrobial property of the threads. The first plate was incubated at $37\text{ }^\circ\text{C}$ under ambient

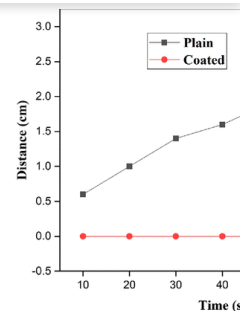


Figure 10. Water penetration rate of pure and MoS_2 -coated lotus fiber threads.

Figure 11

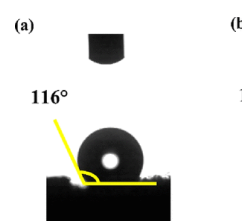


Figure 11. Contact angle measurements of uncoated and MoS_2 -coated lotus fiber threads.

3.1. Structural Studies

XRD analysis of Pure and MoS₂ coated lotus thread was carried out for analyzing its structure. Patterns of pure lotus fibers were well matched with cellulose crystalline standards. Observed XRD reflections of lotus fibers are well-matched with the cotton I β cellulose. The cotton I β cellulose reference pattern was taken from CIF file no. 4114994 using the Mercury 3.8 program. (40) Comparative patterns of experimental (fiber) and calculated (cotton I β cellulose) are depicted in Figure 2. The comparison clearly shows that the obtained major reflection for lotus fiber planes such as (1-10), (110), (102), and (200) were well matched with the calculated one with a broader pattern. The recorded pattern of fiber@MoS₂ is presented in Figure 3 and was compared with the calculated standard with CIF file no. 9007660 of MoS₂, (41) which exhibits a hexagonal structure. The obtained composite pattern clearly shows that the broad pattern at (002) reveals the thin layers of MoS₂ sheets. Mugashini et al. confirm the thin layer of MoS₂ nanosheets. (42,43) The high crystalline peak of MoS₂ at the (100) plane supports the island growth nature of MoS₂.

Figure 2

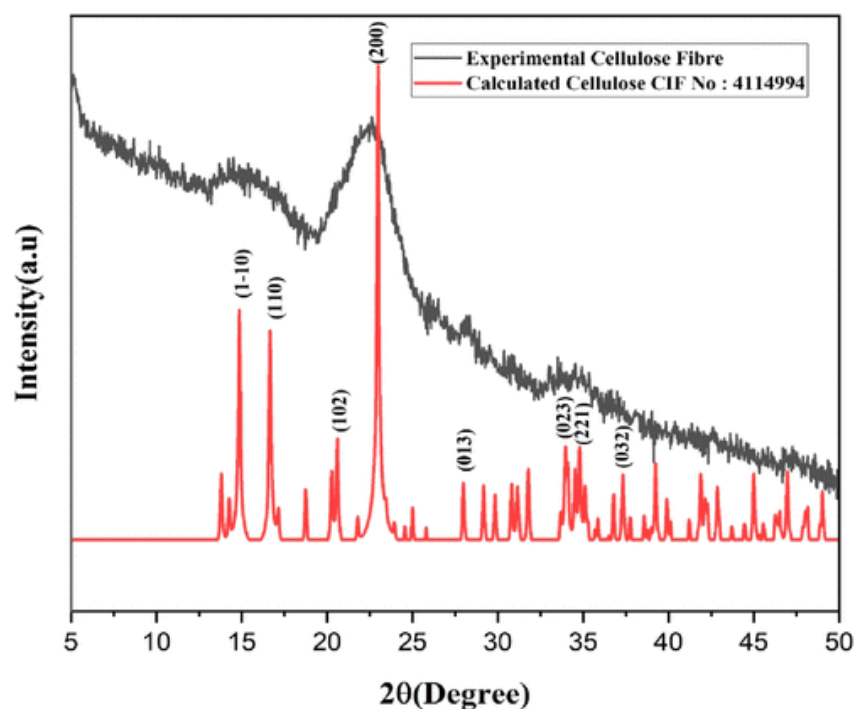


Figure 2. Comparative XRD pattern of lotus fiber and calculated cotton I β cellulose patterns.

Figure 3

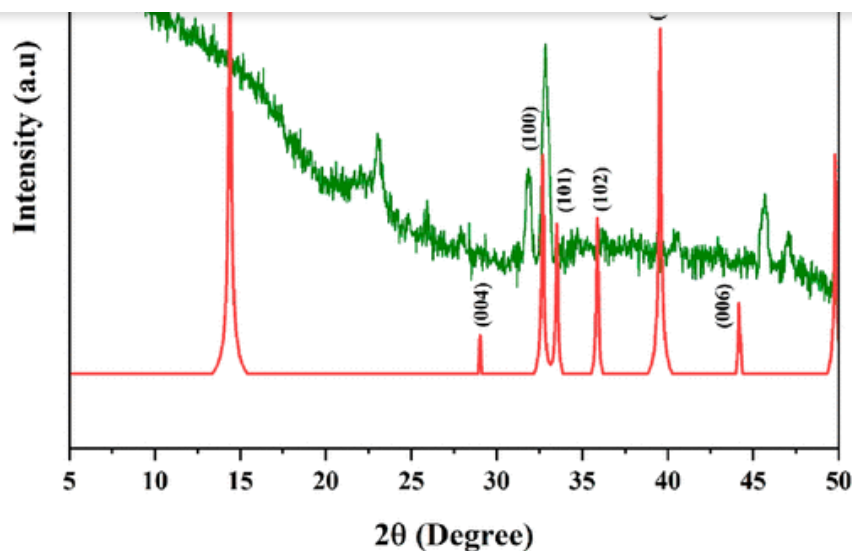


Figure 3. Comparative XRD pattern of MoS₂@fiber and calculated MoS₂.

The sharp intensity patterns on cellulose and fiber@MoS₂ at $2\theta = 14.3^\circ$ and 23° , corresponding to (002) and (200) planes, respectively, are examined. Figure 4 confirms the improved crystalline nature of lotus fibers after acid treatment. The sharp reflection at the (200) plane supports the fiber@MoS₂ containing the crystalline cellulose. (44). Generally, MoS₂ has a sheetlike structure spread on the surface in addition to the island (pitted) growth. The (002) plane appears broader (thin layers of MoS₂), which is due to the sheet structures, and the planes (100), (101), (102), and (103) confirm the island formation.

Figure 4

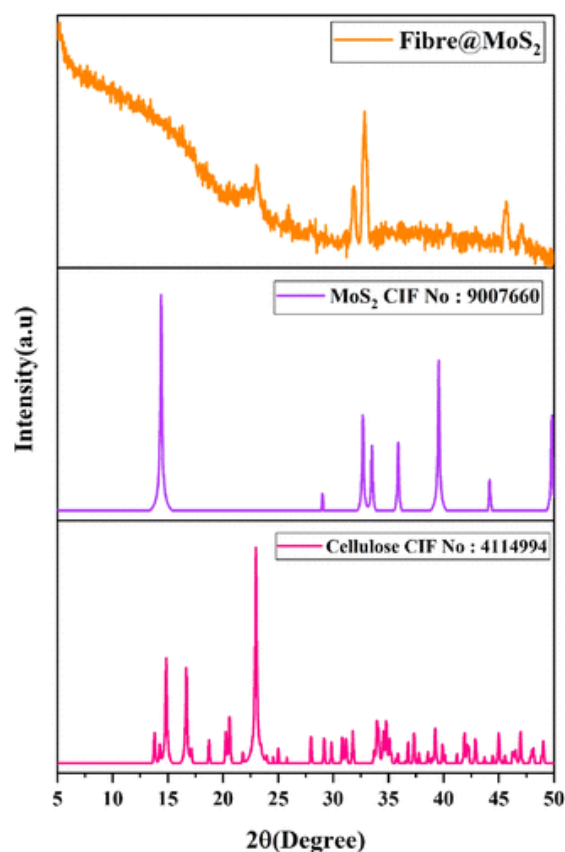


Figure 4. Comparative XRD data of fiber@MoS₂ with CIF File Nos. 4114994 and 9007660 of cellulose and MoS₂, respectively.

and EDX of MoS₂-coated fibers. Excellent moisture absorption and permeability due to the twisted ribbon-like structure were observed. The twisted helical structures of the fibers are observed. With increasing magnification, the H-shaped cuts required for water transportation are visible. Fibers appear slender, and veins are seen in the transverse view of the fiber. The cracks that occurred during fiber extraction are noticed. Damaged areas with cracks result in a fine layer of MoS₂ nanosheets. The nanosheets arise vertically on the fiber's surface, which also appears as H-cuts. The appearance of frequent H-cuts makes fiber water repellent. The diameter of the pure fiber is 2.92 μm, whereas the diameter of fiber@MoS₂ is 2.89 μm.

Figure 5

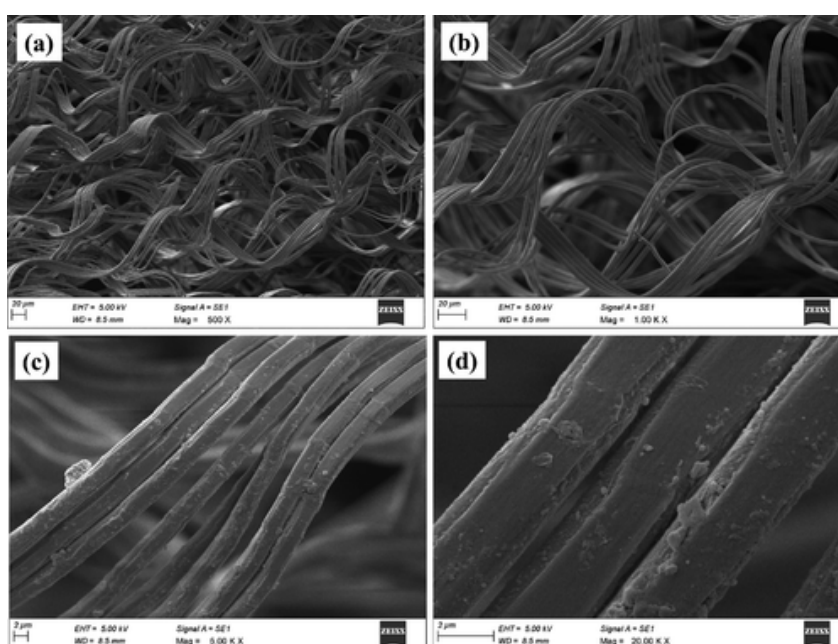


Figure 5. Different magnification SEM images (a–d) of pure lotus fiber.

Figure 6

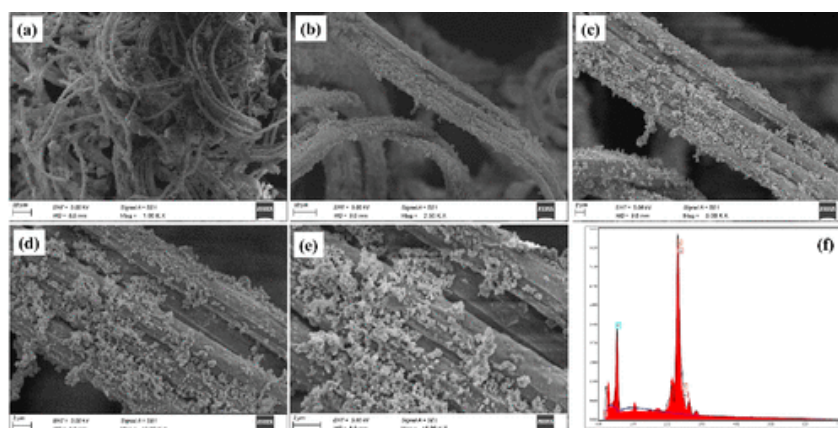


Figure 6. Different magnification SEM images (a–e) and EDX (f) of MoS₂-coated lotus fiber.

3.3. Antifungal Activity

organism cultures, and the zone of inhibition of growth around the discs is studied to determine the antimicrobial property of the test compound. (37,45–47). Similarly, in our study, we have checked for the presence of a zone of inhibition of growth formed around the uncoated and MoS₂ nanosheet coated lotus fiber threads placed on the agar media plates inoculated with *C. albicans* culture and were further incubated at 37 °C. Under ambient light conditions, the MoS₂-coated thread exhibited more antifungal activity than the uncoated or plain thread (Figure 7a). Similarly, the MoS₂-coated thread exhibited more antifungal activity under dark conditions than the uncoated or plain thread (Figure 7b).

Figure 7

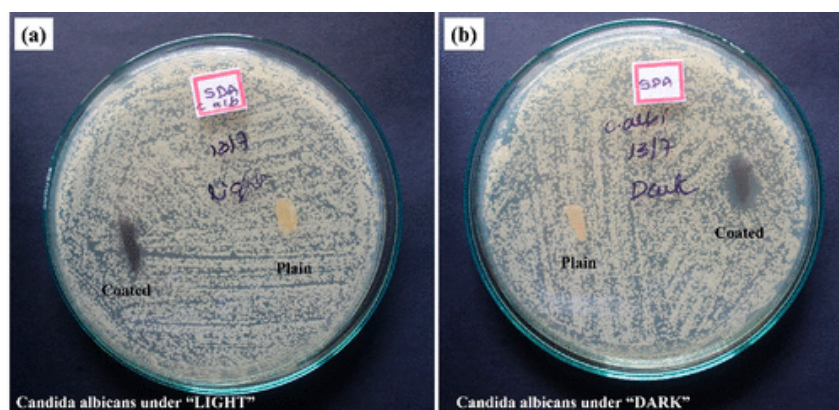


Figure 7. Antifungal activity of pure and MoS₂-coated lotus fiber under light (a) and dark (b) conditions.

Interestingly, the zone of inhibition (ZOI) in the dark was more prominent than in the light experiments. We hypothesize that this may be due to the photosensitive nature of MoS₂ nanosheets in the presence of ambient light and dark conditions. The study confirms that the growth of fungi *C. albicans* around the uncoated or plain lotus threads is attributed to no antifungal activity.

3.4. Antibacterial Activity

Figure 8 represents the antibacterial activity of uncoated and MoS₂-coated lotus threads under ambient light and dark conditions. Antibacterial activity of the MoS₂-coated lotus fiber thread was observed mainly under the dark conditions, depicted by the zone of inhibition of growth of *E. coli* formed around the coated thread. However, significant growth of the organism was observed around the uncoated or plain thread under both ambient light and dark conditions exhibiting no antibacterial activity. Thus, the antimicrobial studies conducted confirm the more antibacterial and antifungal activity of the coated nanoparticle lotus fiber threads under dark conditions than in ambient light. MoS₂ nanosheets can generate ROS and induce physical damage for bacterial inactivation. (48). Similarly, Basu et al. have shown the antifungal and antipollutant activity of MoS₂ nanosheets under dark conditions. (49). In their seminal work, Alimohammadi et al. reported that peptidoglycan mesh in the bacterial cell wall has been indicated as a primary target for interaction with the sheets leading to morphological changes and cell wall damage. (50). A comparative table depicting the antimicrobial activity of MoS₂ by various

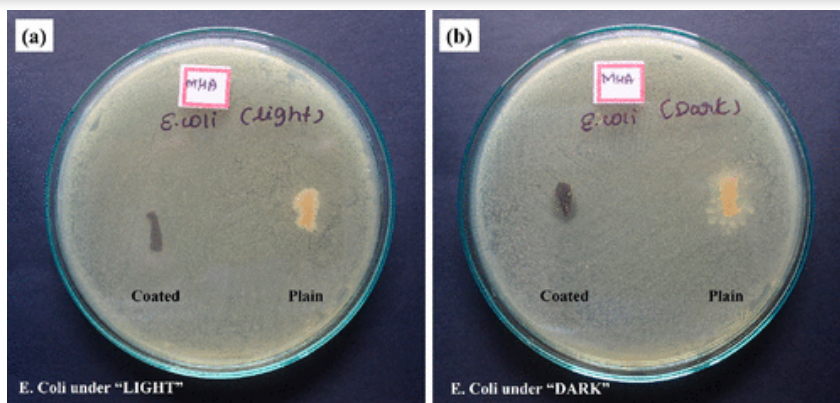


Figure 8. Antibacterial activity of pure and MoS_2 -coated lotus fiber under light (a) and dark (b) conditions.

3.5. Water Absorbance and Penetration Assay

On plain lotus fiber, the water absorbance is relatively high. Liu et al. confirmed the rate of faster absorption of water in the lotus fiber. (53). On the other hand, lotus fiber coated with nanostructured MoS_2 has low absorbance, making it water resistant, which can be potentially integrated with fabrics. Figure 9 shows the water absorbency graph on pure and MoS_2 -coated fiber, which indicates that coated fibers tend to absorb about 80% when compared with the absorption of uncoated fibers. Figure 10 gives the graphical representation for lateral water penetration on plain and MoS_2 -coated fibers. Water penetration on plain lotus fiber increases gradually over a distance of 3 cm for the observed 60 min, whereas the fiber@ MoS_2 shows no penetration, i.e., 0 cm for 60 min. Thus, the water penetration assay confirmed that no penetration occurs in fiber@ MoS_2 .

Figure 9

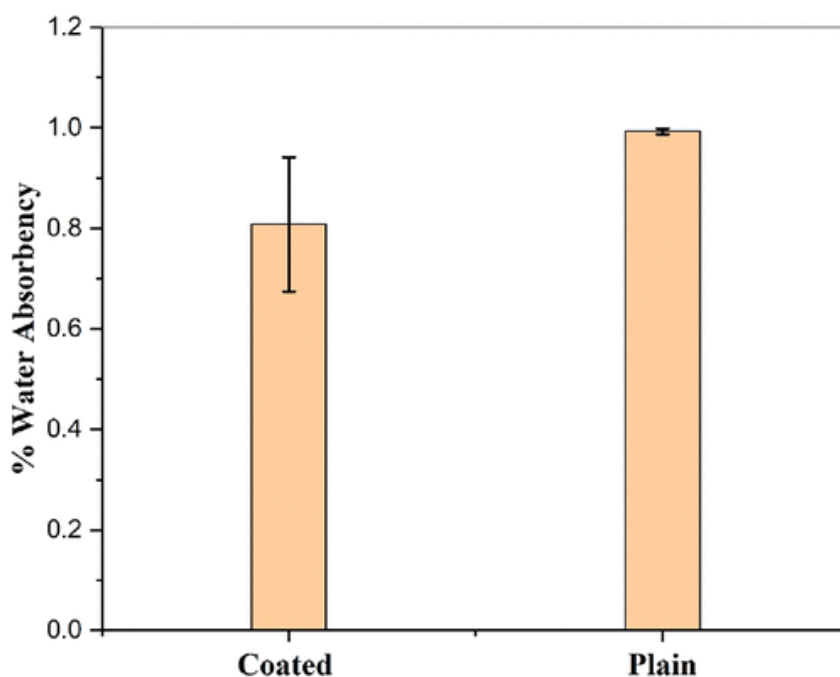


Figure 9. Water absorbency of pure and MoS_2 -coated lotus fiber.

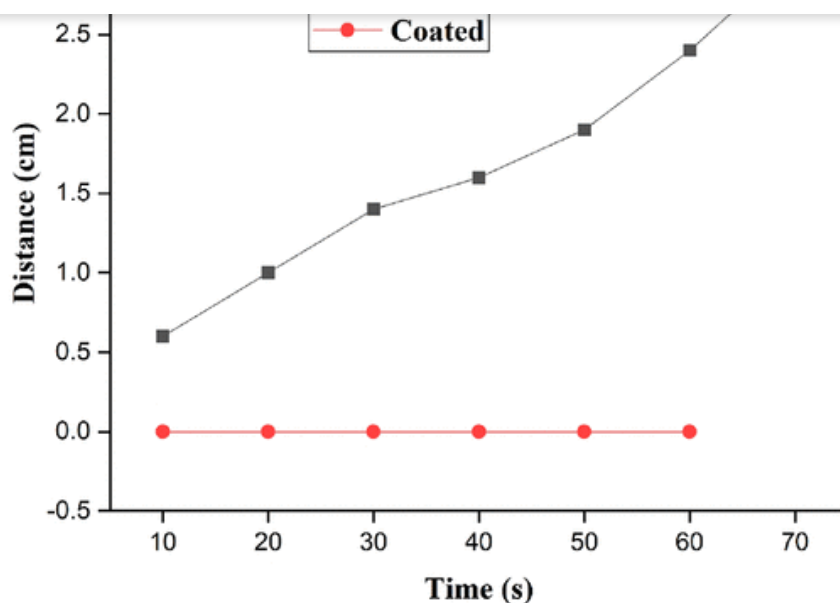


Figure 10. Water penetration assay of pure and MoS₂-coated lotus fiber.

3.6. Contact Angle Measurements

Figure 11 shows the contact angle measurements of pure (a) and MoS₂-coated lotus fiber (b). Observation inferred that pure fiber makes a contact angle of 116°, and MoS₂-coated lotus fiber has a contact angle of 128°, indicating that MoS₂ coating improves the hydrophobicity of fiber. The contact angle value increases toward superhydrophobicity.

Figure 11

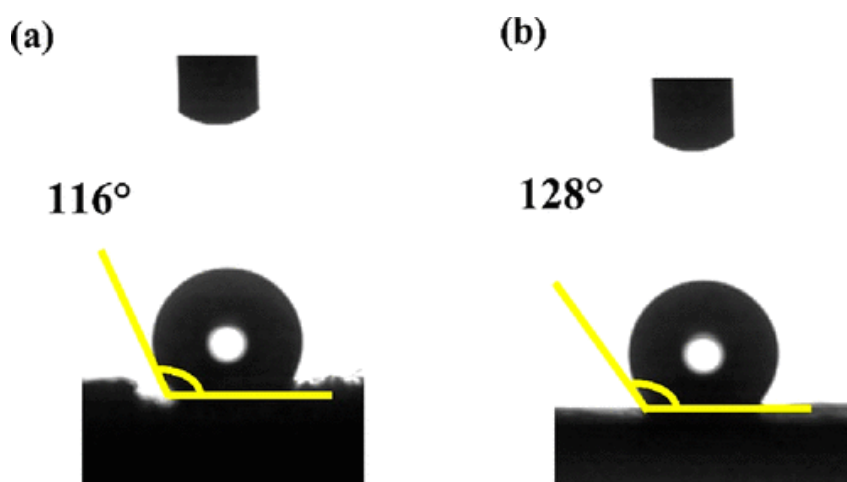


Figure 11. Contact angle of pure (a) and MoS₂-coated fiber (b).

4. Conclusion

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The coprecipitation method was used to assess the hydrophobicity and antimicrobial activity of MoS₂ nanosheets coated on lotus fiber. The XRD patterns confirmed the crystalline nature of pure fiber and fiber@MoS₂. FESEM reveals the morphology of fiber@MoS₂. The growth of MoS₂ nanoparticles over the fiber decreases the wicking ability, confirming the hydrophobic nature of the material. Further, antibacterial and antifungal activities of the MoS₂-coated fiber were verified with *E. coli* and *C. albicans*, respectively. The contact angle of

Supporting Information

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The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.2c02337>.

- Antimicrobial activity of MoS₂ nanosheets (Table S1) (PDF)

Embellishing 2-D MoS₂ Nanosheets on Lotus Thread Devices for Enhanced Hydrophobicity and Antimicrobial Activity

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Embellishing 2-D MoS₂ Nanosheets on lotus thread devices for enhanced hydrophobicity and anti-microbial activity

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Author Contributions

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by S.S. Govarthini, D. Thangaraju, Anusha Prabhu and Naresh Kumar Mani. The first draft of the manuscript was written by S.S. Govarthini and D. Thangaraju, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript. S.S. Govarthini: Writing - original draft. D. Thangaraju: Methodology, Conceptualization, Visualization, Methodology, Supervision, Writing - review and editing. Anusha Prabhu: Methodology, Writing - review and editing. Naresh Kumar Mani: Conceptualization, Visualization, Methodology, Supervision, Writing - review and editing.

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Notes

The authors declare no competing financial interest.

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