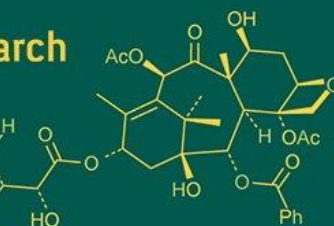
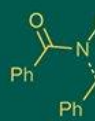


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Sustainable development of fibrous cocoon waste of *Antheraea proylei* and *Bombyx mori* as a sorbent for purification of various oil spills from water

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Abstract

The objective of this study is to evaluate the efficiency of needle-punched silkworm cocoon waste derived from *Antheraea proylei* and *Bombyx mori* as sorbent materials for the removal of oil spills from water. The investigation was conducted using six different types of oils: motor oil, vegetable oil, mustard oil, petrol, diesel, crude oil, and lubricant oil. Key parameters assessed included oil sorption capacity, oil retention capacity, and the reusability of the needle-punched sorbent materials. The results demonstrated that *Antheraea proylei*-based needle-punched sorbents exhibited the highest oil sorption capacity with motor oil, reaching 48.72 g of oil per gram of sorbent. In contrast, *Bombyx mori*-based sorbents showed maximum sorption with crude oil, recording a capacity of 37.29 g/g. Both types of sorbent materials displayed significant oil retention abilities and could be reused effectively up to five cycles without substantial loss in performance. This study highlights the potential of silkworm cocoon waste as a sustainable, eco-friendly, and cost-effective sorbent material for the removal of various oil contaminants from aquatic environments. The findings support the development of value-added applications for sericulture byproducts, contributing to environmental protection and waste valorization.

Keywords: Silkworm, cocoon waste, sorbent, water pollution, oil spill, oil sorption, oil retention

Introduction

Silk is considering as natural protein polymer and known as “Queen of fibers”. Silkworm larvae spun silk, which is widely used in textiles. During spinning of cocoon, silkworm larvae produce silk to form protective shell for delicate caterpillar. Pupal stage is pass inside the cocoon and metamorphose into the adult stage (Bentley & Auldrige, 2017) [2]. These cocoons are considered as commercial sources of silk material. Silk materials have outstanding mechanical strength and toughness. Silk is considered bio-compatible and biodegradable. Silkworm evolved cocoon for protection against various threats and to regulate gaseous exchange for pupa development. Silkworm cocoon is a non-woven structure, and it is made up of continuous silk fibers joined together by sericin matrix. For commercial purpose these silkworm cocoons are cultivated to yield silk fibers in apparel and biomedical applications (Bucciarelli & Motta, 2022) [3]. In India, commonly two types of silkworms cultivated namely *A. proylei* and *B. mori*. Release of oil spills into water continuously affecting aquatic ecosystem, oil spills have various hazardous compounds which enters by means of food chains, leading to severe health issues in humans. Now a days biodegradable cocoon waste acts as oil sorbent gaining too much attention due to their ecofriendly nature. Recently many researchers investigated the unique property of silk fibers for oil water separation (Gore *et al.*, 2020; Yin *et al.*, 2022) [7, 6].

Sorbents have the ability to concentrate and alter them into semisolid state, and spilled oil can be easily removed by convenient methods (Moriwaki *et al.*, 2009) [8]. Presently many natural biodegradable sorbents such as wool fibers, cotton fibers, collagen fibers and kapok fibers have been used as sorbent to clean up leaked oil (Moriwaki *et al.*, 2009; Subramoniapillai & Thilagavathi, 2022) [8, 10]. These sorbents have good sorption capacity, low cost, renewable and biodegradable. Soil and silicates are also currently used to clean up

various oil spills, but it takes lot of energy and cost to clean up leaked or spilled oil. To solve this problem this study used sorbents derived from fibrous cocoon waste. Application of natural silk fibroin waste is very important to study (DeBari *et al.*, 2021; Nogueira *et al.*, 2010) [4, 9]. Hydrolyzed fibroin (HF) from the fibrous silkworm cocoon waste can be used as an efficient sorbent for the purification of various oils in automobile industries, food industries, in pharmacy and textile industries (Viscusi, 2023) [14]. During the extraction of silk fiber from the silkworm cocoons (reeling) up to 28% of silk fibers are converted into waste (depends upon the silk spinning mill). In terms of quality and mechanical properties silk fiber waste is considered as inefficient for textile industries. Whereas chemical properties of both silk fibers and fibrous waste are same. That is why hydrolyzed silk fibroin has excellent sorption property, and this property can solve various problems practically. Silk fibers consist of fibroin and sericin proteins, the amount of these proteins in the silk fibers depends upon the quality of diet and living conditions of worms. The present study aims to investigate the potential application of fibrous cocoon waste, a byproduct remaining after the reeling process, as a sorbent material for various types of oils. Emphasis is placed on evaluating the oil sorption behavior of this fibrous waste, with the objective of exploring its effectiveness and suitability as a sustainable and low-cost sorbent for oil spill remediation and related environmental applications.

Materials and Methods

Silkworm cocoon waste of *Antheraea proylei* was procured from the Sericulture Research Extension Center, Chauntra, Mandi, Himachal Pradesh, India, while cocoon waste of *Bombyx mori* was obtained from the Silkworm Seed Production Center, Palampur, Himachal Pradesh, India. Six different oil samples, namely mustard oil, petrol, diesel, crude oil, motor oil, and lubricant oil, were used in this study and were purchased from the local market. The density and viscosity of the oils were measured at room temperature. The density of mustard oil was 0.92 g/mL, with a viscosity ranging from 60 to 70 mm²/s. Petrol exhibited a density between 0.72587 and 0.7429 g/mL and a viscosity of 0.88 mm²/s. Diesel had a density range of 0.83 to 0.86 g/mL and a viscosity range of 2.0 to 4.5 mm²/s. Crude oil displayed a density between 0.70 and 0.99 g/mL, with viscosity ranging from 2 to 10 mm²/s. The density of motor

oil ranged from 0.866 to 0.89 g/mL, with viscosity values between 43 and 60 mm²/s. Lubricant oil showed a density of 0.866 g/mL and a viscosity range of 55 to 70 mm²/s.

- **Sorption analysis:** Six samples of oil were used for the experiment namely motor oil, vegetable oil, mustard oil, petrol, diesel, crude oil, and lubricant oil. One gram of oil in a Petri dish was placed for 24 hours and 10 days at room temperature. Then, the weight of oil loss was measured by an analytical balance.
- For oil sorption and retention capacity 10 gm of motor and vegetable oil were added to the beaker containing 100 mL of distilled water for the sorption experiment. Therefore, after 10 minutes, 0.1 g of the SCWS was generously applied to the oil surface. The material was then removed using forceps and allowed to drain for one minute. Oil-sorbed material was placed on a petri plate. In comparison to that dried for 10 days at room temperature, drying for 24 hours had no effect. So, a 24-hour drying window was established as shown in **Figure 1**. The oil-sorbed material was weighed using an analytical balance after drying. The testing was done both with and without stirring (S Viju *et al.*, 2019) [11].
- **Reusability test analysis:** For the material's reusability test, 10 gm of motor and vegetable oil were added to a beaker with 100 ml of distilled water, and 0.1 gm of SCWS was then sprinkled on top of oil. For each sample, the Sorption process was run five times. The material was weighed and squeezed in between each cycle. The amount of oil recovered was determined by dividing the difference between the material that had been squeezed and that which had been previously sorbed with oil.
- **Statistical analysis:** Statistical analysis and plotting of graphs were done using Origin Pro 2025 (graphing and analysis) and Microsoft Excel.

Results

For oil spill clean-ups, it is crucial to consider the sorbent material's oil sorption and oil retention capabilities throughout field application, handling, and transfer. The sorbent material made of silkworm cocoon waste was water repellent. The oil sorption and oil retention performance of the sorbent immediately and after 24 hours is shown in Figure 1 (a & b).

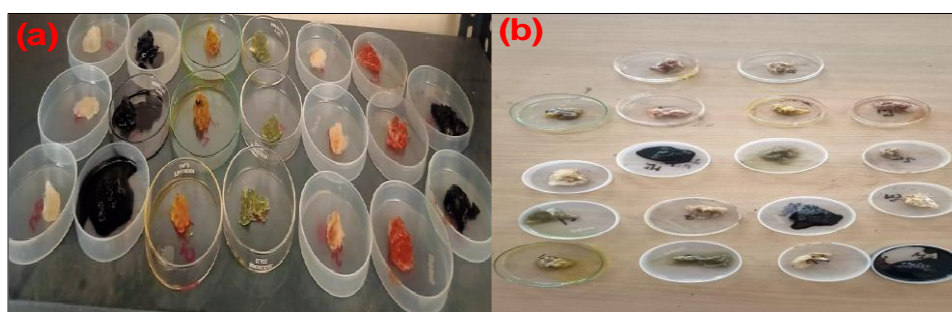


Fig 1: (a) The sorbent ability of silkworm cocoon waste sorbent (SCWS), (b) the oil retention capacity of cocoon waste sorbent after 24 hours.

Calculating the oil sorption capacity of sorbent material is considered as one of the important criteria for oil spill cleanup. The oil sorption capacity needle punched sorbent

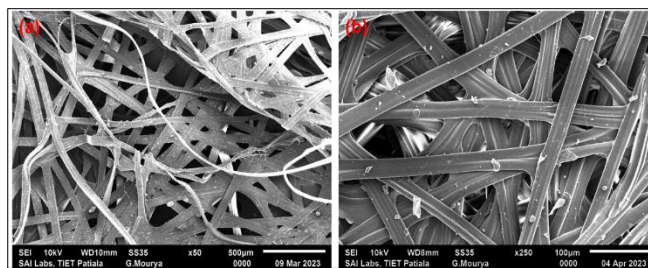
material produced from *A. proylei* and *B. mori* silkworm cocoon waste was calculated and compared presented in Table 1.

Table 1: Showing the oil sorption ability of silkworm cocoon waste.

Name of Oil	<i>Antheraea proylei</i> (g/g)	<i>Bombyx mori</i> (g/g)
Mustard oil	37.21	30.69
Petrol	18.85	14.56
Diesel	23.79	15.97
Crude oil	42.28	37.29
Motor oil	48.72	30.62
Lubricant oil	41.77	28.53

The highest oil sorption capacity in *A. proylei* needle punched sorbent material (cocoon waste) with motor oil 48.72 g/g and lowest with petrol 18.85 g/g. The highest oil sorption capacity in *Bombyx mori* needle punched sorbent material (cocoon waste) with crude oil 37.29 g/g and lowest with petrol 14.56 g/g. The oil sorption capacity mainly depends on the structural characteristics of fibers. The above results indicate that oil sorption capacity of *A. proylei* cocoon wastes, needle punched sorbent material is higher than that of *B. mori*. (S Viju *et al.*, 2019) ^[11] mentioned in their research that high viscous oil showed higher adsorption than low viscous oils. Similarly present research also concludes that motor oil and crude oil highly absorbed by needle punched fibers of silkworm cocoon wastes than vegetable oil and petrol. The reason could be higher viscosity of motor oil and crude oil than vegetable oil and petrol. In general, high viscous oils show higher adsorption capacity than low viscous oils because of capillary diffusion of oils into the pores present in the fiber's structures leading to the higher adsorption capacity (S. Viju *et al.*, 2019) ^[11].

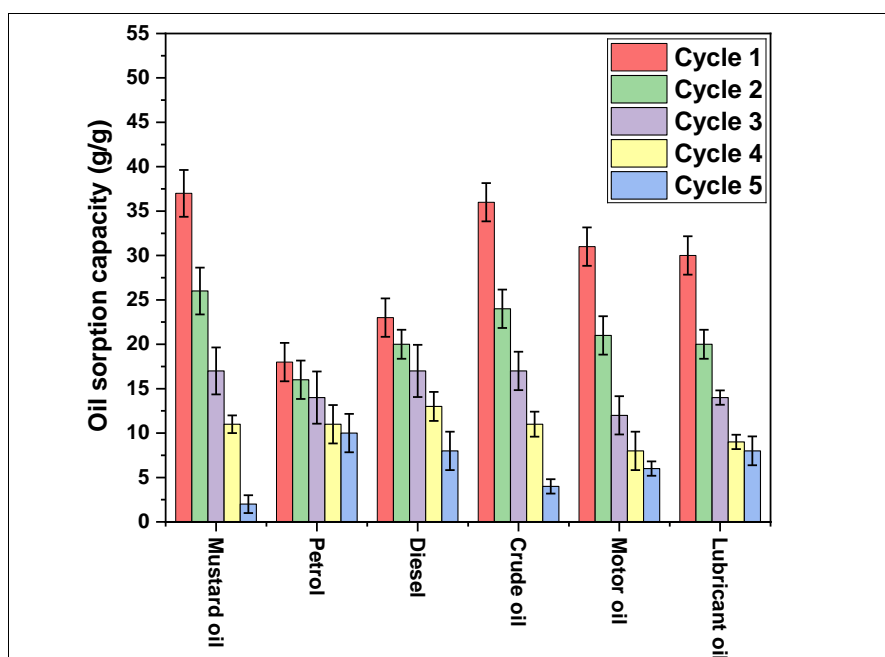
Hydrophobic amino acid such as alanine, phenylalanine, tryptophan, valine, methionine and proline are mainly present in silk, mainly responsible for hydrophobic-oleophilic nature of the silk. (Aftabuddin & Kundu, 2007; Gore *et al.*, 2019) ^[6] reported that silkworm raw silk fibers composed of 70% to 80% fibroin, 20-30% sericin, 1.2% to 1.65% carbohydrates, 0.4% to 0.8% waxy matter, 0.7% inorganic matter, and 0.2% pigment. SEM micrographs of *A. proylei* and *B. mori* cocoon structure displayed in Figure 2 (a & b).

**Fig 2:** SEM micrographs of (a) *Antheraea proylei* cocoon (b) *Bombyx mori* cocoon.

Oil retention refers to the amount of oil that a sample can absorb per unit of weight Table 2. The oil retention property of needle punched sorbent material of *A. proylei* highest in crude oil 65.67% and lowest in petrol 48.13%. The oil retention property in the case of *B. mori* sample highest in crude oil 68.29% and lowest in diesel 46.45%. Furthermore, in the samples of both species crude oil showed the highest oil retention ability compared to the rest of the samples. The oil retention ability of needle punched sorbent material of *A. proylei* and *B. mori* showed variation in different liquid oils may be due to the different adhesive properties between the oil and fibrous material of cocoon waste. The variation in the properties of adhesiveness is linked with the viscosity of each liquid oil sample. (Dong *et al.*, 2015) ^[5] discussed in their research that desorption of oils in needle punched sorbent material of cocoon waste is slower in case of high viscous oil as compared to low viscous oil. According to the results crude oil showed a slower desorption process due to its higher viscous nature.

Table 2: The oil retention property of silk cocoon waste.

Name of oil	<i>Antheraea proylei</i> (%)	<i>Bombyx mori</i> (%)
Mustard oil	58.35	56.92
Petrol	48.13	52.85
Diesel	60.83	46.45
Crude oil	65.67	68.29
Motor oil	53.29	58.59
Lubricant oil	65.09	48.65

**Fig 3:** The reusability of silk cocoon waste as a sorbent (*Antheraea proylei*).

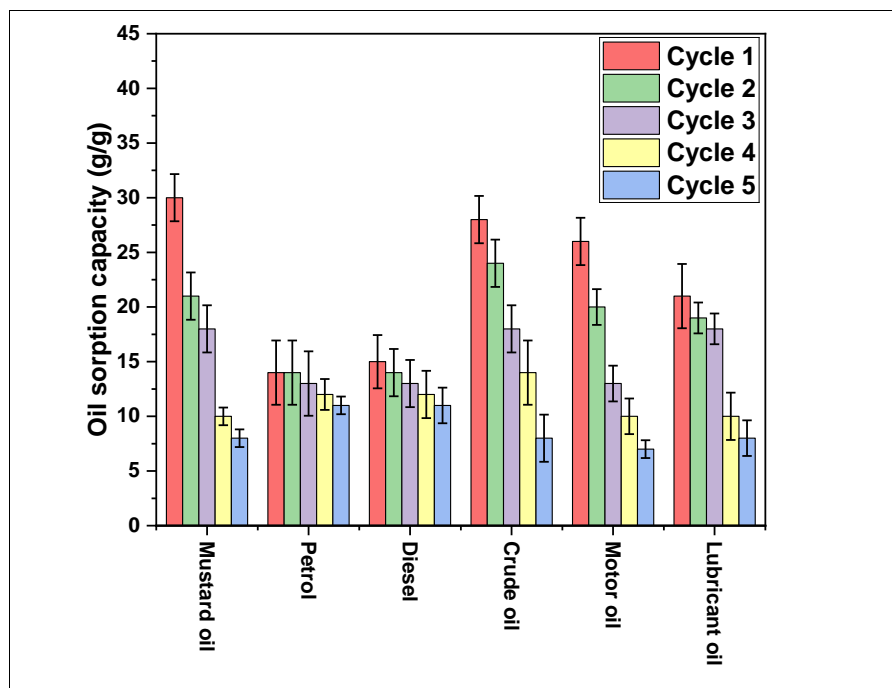


Fig 4: The reusability of silk cocoon waste as a sorbent (*Bombyx mori*).

Discussion

One of the key characteristics of oil sorbent material is its capacity to be reused. The price of managing oil pollution can be decreased by using materials that can be recycled frequently. Figures 3 and 4 display the oil sorption performance of silk cocoon waste for five cycles of sorption and desorption cycles for both species of cocoon waste samples. After five sorption-desorption cycles the oil sorption capacity sorbent material of *A. proylei* cocoon waste after five sorption-desorption cycles were 4.0 g/g, 2.0 g/g and 8.0 g/g against crude oil, mustard oil and lubricant oil. The oil sorption capacity sorbent material of *B. mori* cocoon waste after five sorption-desorption cycles were 8.0 g/g, 8.0 g/g and 7.0 g/g against mustard oil, crude oil and motor oil. The oil sorption capacity of needle punched sorbent material continuously decreases with every cycle. The reason could be tightening of inter fiber spaces, irreversible deformation of few fiber structures or incidence of residual oils. (S. Viju *et al.*, 2019) [11] proved in their research that after each cycle, the oil sorption ability of silkworm cocoon waste continues to decline. (Wang *et al.*, 2012) [15] has also discussed in their research that, presence of residual oil in nonwoven fabrics, irreversible deformation of partial fiber structure, and tightening between inter fiber gaps are few potential causes for the decrease in oil sorption capacity with every cycle.

The sorbent material was made from residual silkworm cocoons (SCWS), and the SCWS greatly varies from how quickly it absorbed different oils. By subtracting the starting sorbent weight from the total weight of the wet sorbent, the amount of oil that was absorbed was calculated. The porosity, specific surface area, and pore estimate of the sorbents were strongly correlated with differences in oil sorption capacity. As oil consistency increases, sorption capacity rises. The variation in oil viscosities was primarily responsible for the variation in adhesiveness. High-viscous oils desorb from nonwoven textiles more slowly than low-viscous oils during the draining stage (Dong *et al.*, 2015) [5]. It is evident that after each cycle, the silk non-woven fabric's

ability to absorb oil continues to decline. this may be caused by a number of things, such as the presence of residual oil in nonwoven fabrics, tightening of inter-fiber gaps, and irreversible deformation of partial fiber structure (Wang *et al.*, 2012) [15]. In order to assess the material's oil sorption capability, oil retention capacity, and reusability silkworm cocoon waste was used as a sorbent material. *B. mori* has a lower oil sorption capacity than *A. proylei*, indicating that the latter is preferable in terms of sorption capacity, albeit each oil sample's effect on each cocoon's oil retention capacity will vary. Continuously using the cocoon waste sample for five cycles reveals a consistent decline in oil sorption capacity for both cocoons. There is no residue of silk cocoon waste found after 100 days of burial in soil (S. Viju *et al.*, 2022) [13]. According to the results of this study, the silk cocoon waste from *A. proylei* has greater sorbent activity than *B. mori*. The silk cocoon waste with good sorption capacity can be used for the solution of two problems, i.e., purification of oils and reducing oil pollution in natural waters.

Conclusion

In conclusion, this research shows that silk cocoon waste from *Antheraea proylei* and *Bombyx mori* exhibits effective oil sorption, retention, and reusability characteristics. *A. proylei* showed better sorption capacity, specifically with high-viscosity oils like motor oil, because of its beneficial fiber structure. Crude and motor oils were more efficiently absorbed and retained than low-viscosity oils such as petrol. Amino acid composition played an important role in oil affinity, and it is attributed to the hydrophobic-oleophilic nature of silk. Reusability analysis showed a gradual decline in performance over 5 cycles which may be due to residual oil accumulation and fiber structure changes. A 24-hour drying period was found adequate, with no negative impact on sorption ability. Notably, *B. mori* exhibited slightly better oil retention in crude oil, although it was found that overall, *A. proylei* was more efficient. The SCWS was also confirmed to be biodegradable, which left no residue after

100 days of burial in the soil. Therefore, SCWS, particularly from *A. proylei*, is a promising, eco-friendly, and reusable material for oil spill remediation and purification applications.

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