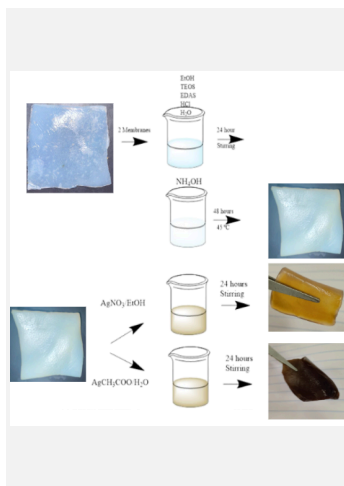


## Hybrid Silver-Loaded Bacterial Cellulose/Organosilica Photocatalytic Aerogels for In-Flow Water Purification

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Aerogels, known for their high porosity and low density, have notable adsorption capabilities and serve as supports for catalysis, but they lack mechanical strength. To reduce this fragility, hybrid organic-inorganic materials such as the combination of bacterial cellulose (BC) with organosilica can be developed, which can be applied in water purification. In this research, five BC/Organosilica hybrid materials were developed using sol-gel synthesis and precipitation, with varying proportions of TEOS and EDAS. Silver ions were incorporated to add photocatalytic properties by in-situ formation of  $\text{Ag}_2\text{O}$ . After preparation, the materials were characterized and their photocatalytic properties were tested to degrade methylene blue in a in-flow membrane photoreactor. The hybrid 70% TEOS/30% EDAS modified with silver acetate showed the best results, removing 74% of the methylene blue. This hybrid materials approach offers a promising solution to strengthen aerogels and expand their practical applications, particularly in water purification.

### Introduction

Aerogels are notable for their high porosity and low density, making them highly efficient in various applications such as adsorption, catalysis, and catalyst support. However, their extreme porosity and low density can lead to mechanical fragility, limiting their practical applications. Therefore, there is a need to develop stronger materials.<sup>1,2</sup>

A promising solution is the production of organic-inorganic hybrid materials. Bacterial cellulose (BC) is a strong candidate for creating hybrid materials with inorganic aerogels due to its stable matrix, high surface area, and ease of production.<sup>3</sup>

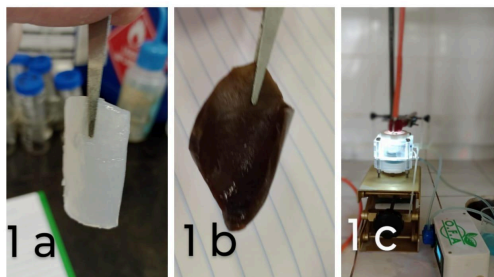
To improve water purification performance, five BC/Organosilica hybrid materials were developed using sol-gel synthesis and precipitation. Precursors such as Tetraethyl Orthosilicate (TEOS) and N-[3-(trimethoxysilyl)propyl]-Ethylenediamine (EDAS) were used in different proportions, and silver ions were incorporated for photocatalytic properties.

### Material and Methods

Firstly the bacterial cellulose matrix was cut into 3.5 cm x 3.5 cm pieces. Then, the pieces were placed in an ethanol bath for a week, with daily ethanol changes to remove synthesis residues. After cleaning, two membranes are placed in a Falcon tube, and 260 mmol of ethanol, 0.6 mmol of HCl, and 40 mmol of water are added to the tube. The quantities of TEOS and EDAS were added according to Table 1. The tube is kept at room

temperature with constant stirring for 24 hours to allow penetration into the membrane and the initial hydrolysis of the silane precursors. Then, 2.14 mmol of ammonium hydroxide were added, and the tube is kept at 45°C for 48 hours. The modified membranes can be seen in Figure 1a.

After the organosilica was added to the bacterial cellulose matrix, the membranes underwent another wash in an ethanol bath for one week, with daily solvent changes. To confer photocatalytic properties, 0.5 mmol of silver acetate was added to 1400 mmol of water, or 0.5 mmol of silver nitrate was added to 428 mmol of ethanol. After the addition of the silver ions, the solution was maintained at room temperature for 24 hours with constant agitation, Figure 1b. Then, the membranes were washed and dried using supercritical drying.



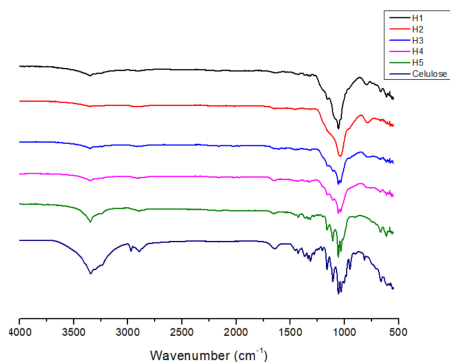
**Figure 1.** 1a) Membrane modified with organosilica; 1b) Membrane modified with silver acetate; 1c) Photo-catalysis system in flow.

Finally, the membranes were characterized with CHN, BJH, and IR analyses and then used for water purification in flow, as illustrated in Figure 1c.

## Results and Discussion

After the addition of the organosilica, the materials were dried and characterized using different analytical methods. The BJH technique was used to determine the surface area, while the CHN analysis was used to identify the fixation, mainly of nitrogen. Infrared analysis (Figure 2) was also carried out. The H3 hybrid, see table 1, was selected for showing the best results in the combination of these characterization methods. The H3 hybrid showed a significant increase in surface area, from 80 m<sup>2</sup>/g to 98 m<sup>2</sup>/g. Additionally, the nitrogen percentage increased from 0.21% to 4.15%, which is notable, considering the ethylenediamine group is crucial for the adsorption of silver by the matrix. In-situ formation of Ag<sub>2</sub>O mediated by ethylenediamine groups was indicated by gels color change upon contact with silver ions solution only in the materials modified with EDAS. Regarding the flow photocatalysis tests, an experiment was conducted using a 7.5 ppm solution of methylene blue for 30 minutes without the presence of light to minimize adsorption. After this dark period, the solution was exposed to light for an additional 60 minutes. The results revealed a removal of 74% of the remaining dye after the dark period.

This performance indicates that the Silver modified-H3 hybrid aerogel is effective in removing pollutants through in-flow photocatalysis. The additional nitrogen presence, derived from the ethylenediamine group, contributes to a better interaction with silver ions, increasing inorganic photocatalytic loading and the efficiency of the photocatalytic process. This demonstrates the potential of these hybrid materials for water treatment applications, where effective removal of contaminants is essential.



**Figure 2:** Infrared spectra of membranes modified with organosilica

**Table 1.** Composition of Hybrids

Hybrid	H1	H2	H3	H4	H5
TEOS (mmol)	5.0	4.3	3.5	2.0	-
EDAS (mmol)	-	0.8	1.5	3.5	5.0
% N	0.21	3.48	4.15	2.82	0.30

## Conclusions

This study demonstrates the potential of bacterial cellulose/organosilica hybrid materials for water purification through photocatalysis. The approach used to combine the beneficial properties of bacterial cellulose with organosilica resulted in a material with a higher surface area and higher number of ethylenediamine chelating groups, enhancing its effectiveness in silver oxide incorporations. Photocatalysis tests conducted with the H3 hybrid showed a significant photocatalytic removal of methylene blue dye as model pollutants, confirming the efficiency of these materials in water purification. Thus, the hybrid materials developed in this study offer a promising solution to overcome the mechanical limitations of inorganic aerogels, providing an effective and practical alternative for water treatment in flow systems.

## Acknowledgments

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