

# Microwave-Assisted Synthesis of Al<sup>3+</sup>-Modified Posidonia Oceanica Fibers for Sustainable Methyl Orange Removal

Rania Hrichi\*, Ridha Touati\*\*, Aida Kessraoui\*

\* Laboratory of Energetics and Materials (LABEM), High School of Sciences and Technology of Hammam Sousse, University of Sousse, BP 4011 Sousse, Tunisia

\*\* NANOMISENE Laboratory, LR16CRMN01, Centre for Research on Microelectronics and Nanotechnology (CRMN), University of Sousse, Tunisia



## Introduction

The discharge of dye-containing wastewater from industrial activities represents a serious environmental problem due to its adverse effects on aquatic ecosystems. Among synthetic dyes, Methyl Orange (MO) is widely used and difficult to remove by conventional treatment methods. Biosorption using natural and renewable materials has emerged as an efficient and sustainable alternative for wastewater purification. Posidonia oceanica (P.O), an abundant Mediterranean marine biomass, has attracted increasing interest as a low-cost biosorbent. In this study, raw P.O fibers were modified with Al<sup>3+</sup> using a microwave-assisted synthesis method to enhance their adsorption properties. The prepared materials were characterized and evaluated for the removal of MO from aqueous solutions.

### Synthesis of P.O–Al Hybrid Material

P.O fibers were washed, dried, ground, and sieved to a particle size of 125 μm. The fibers were then mixed with an aluminum nitrate solution, and the pH was adjusted to 10. The mixture was subjected to microwave irradiation (511 W, 2 min), followed by washing and drying to obtain the P.O–Al hybrid material.

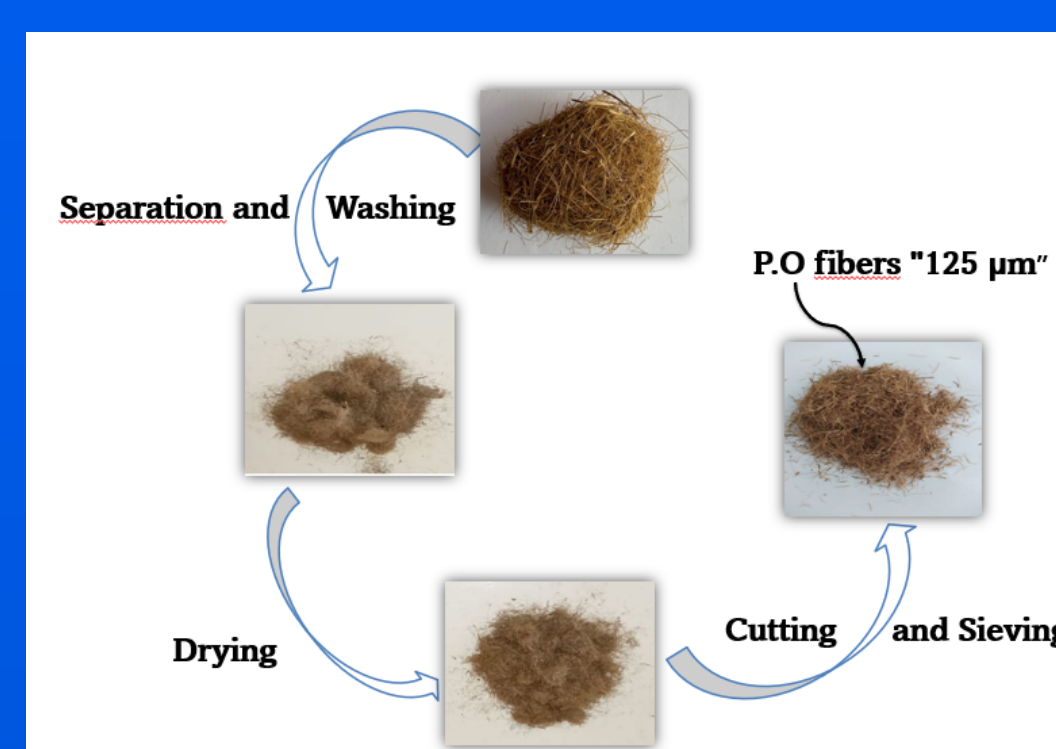


Figure 1: Steps for Processing P.O Fibers



Figure 2: Schematic Illustration of the Microwave-Assisted Synthesis of Al<sup>3+</sup>-Modified P OFibers

## Results & Discussion

### pHpzc and Boehm Titration

Compared with raw P.O fibers (pHpzc = 7.36), Al<sup>3+</sup>-modified fibers prepared by microwave-assisted synthesis showed higher pHpzc values, indicating an increase in surface basicity. Boehm titration revealed a decrease in acidic groups and an increase in basic groups after Al<sup>3+</sup> incorporation. The highest basicity was observed for P.O–4% Al<sup>3+</sup>, confirming the effective interaction of aluminum species with the fiber surface. These modifications promote the adsorption of negatively charged pollutants such as MO.

Table 1 . Surface functional groups of different "P.O-Al<sup>3+</sup>" adsorbents

Adsorbents	Acidic Function (10 <sup>-4</sup> mmol g <sup>-1</sup> )				Basic Function (10 <sup>-4</sup> mmol g <sup>-1</sup> )	pH <sub>pzc</sub>
	Carboxylic	Lactonic	Phenolic	Carboxylic		
P.O -1% Al <sup>3+</sup> MW	0.394	0.402	0.824	1.337	1.673	7.89
P.O -2% Al <sup>3+</sup> MW	0.387	0.376	0.813	1.576	1.941	8.04
P.O -4% Al <sup>3+</sup> MW	0.314	0.299	0.749	1.362	2.128	8.08

### Effect of Al Modification on MO Removal

The MO removal efficiency increased from 51.20% for raw P.O fibers to 53.93%, 59.53%, and 64.89% for P.O–1% Al<sup>3+</sup>, P.O–2% Al<sup>3+</sup>, and P.O–4% Al<sup>3+</sup>, respectively. The progressive increase in removal efficiency with Al<sup>3+</sup> loading demonstrates the positive effect of aluminum incorporation on the adsorption performance of P.O fibers.

The highest MO removal was achieved with P.O–4% Al<sup>3+</sup> prepared by microwave-assisted synthesis. These results justify the selection of P.O–4% Al<sup>3+</sup> prepared by MW for further structural characterization and adsorption modeling.

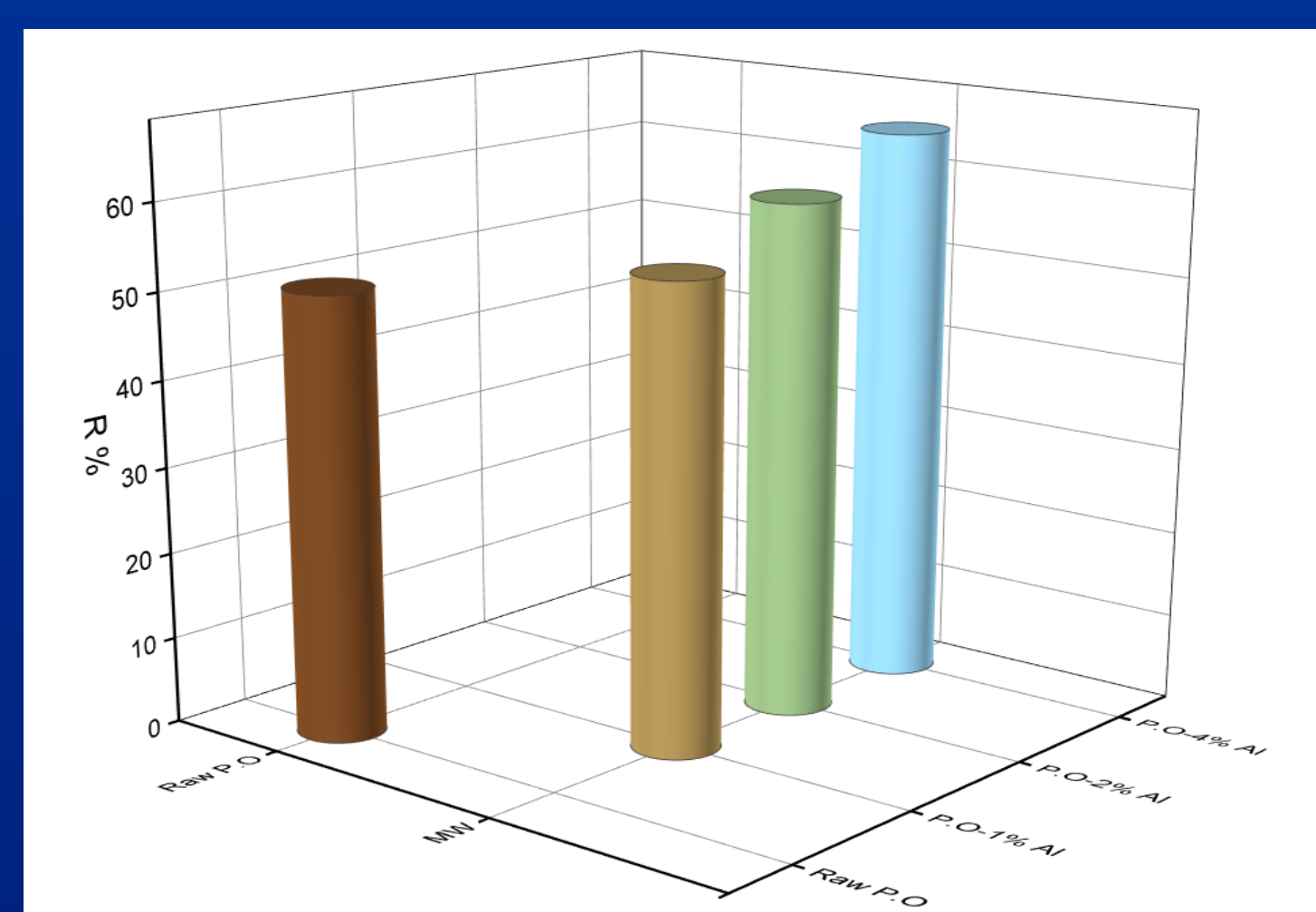


Figure 3: MO Removal Efficiency (%)

➤ P.O–4% Al<sup>3+</sup> showed the highest MO removal efficiency (64.89%)

### SEM–EDX Analysis

SEM images showed that raw P.O fibers possessed an irregular and porous surface. After Al<sup>3+</sup> modification by microwave-assisted synthesis, the surface became rougher and more uniformly covered. EDX analysis and elemental mapping confirmed the successful incorporation and homogeneous distribution of aluminum on the fibers, indicating the creation of new active sites favorable for Methyl Orange adsorption.

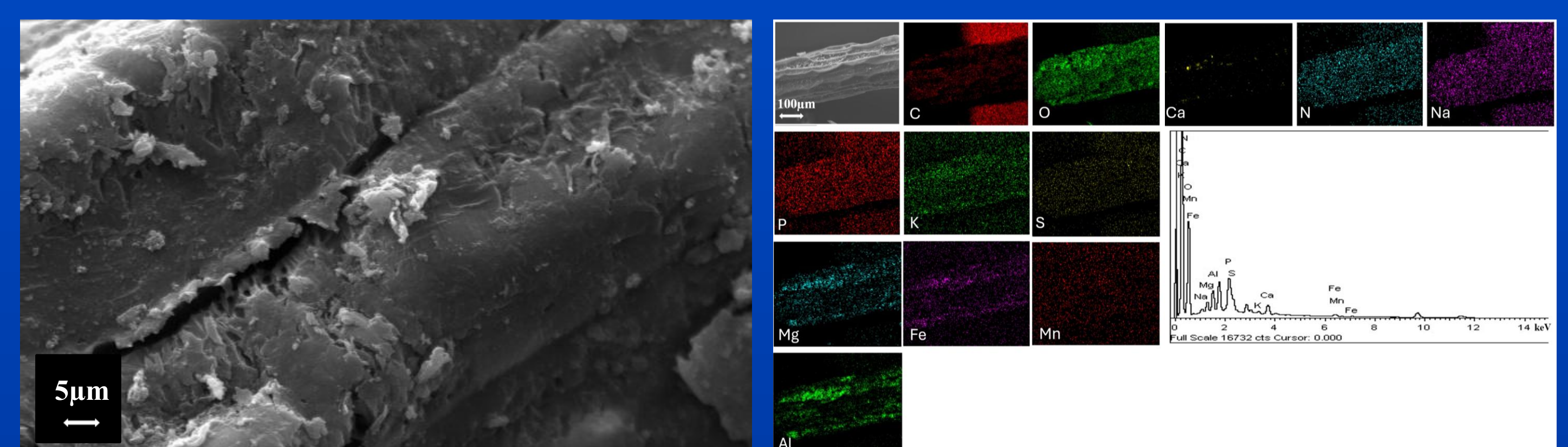


Figure . SEM, EDX and Mapping of P.O-4%Al<sup>3+</sup>+MW

### FTIR and XPS Analysis

FTIR and XPS analyses confirmed the successful incorporation of Al<sup>3+</sup> onto P.O fibers after microwave-assisted modification. FTIR spectra showed changes in the main functional groups and the appearance of an Al–O band, while XPS spectra confirmed the presence of Al–O bonds and oxidized aluminum species (Al<sup>3+</sup>/Al<sub>2</sub>O<sub>3</sub>). These results demonstrate the effective surface modification of P.O fibers and the creation of additional active sites for MO adsorption.

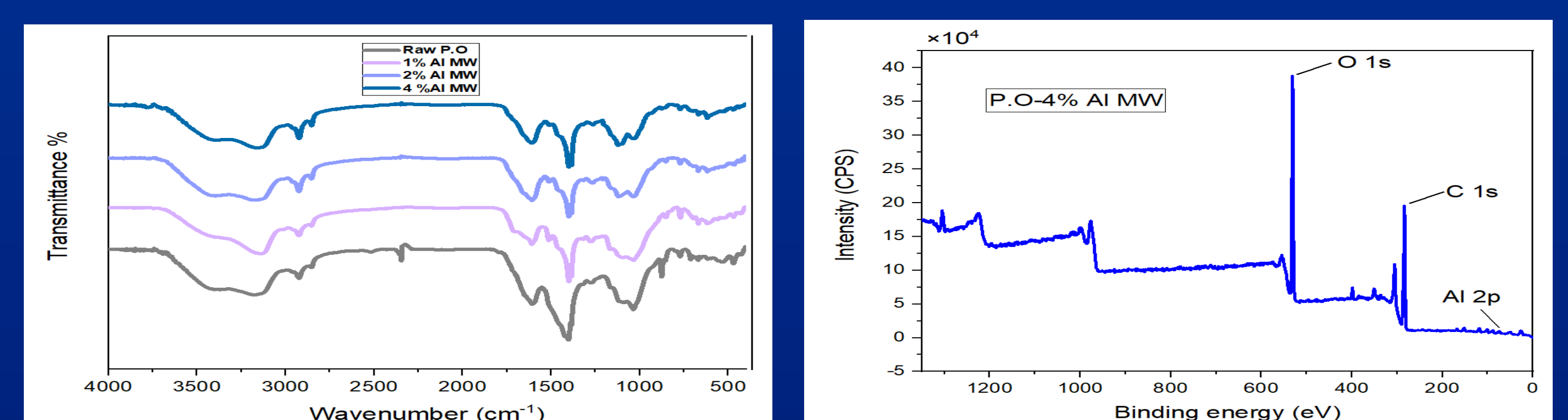


Figure . FTIR and XPS Analysis of P.O-4%Al<sup>3+</sup>+MW

## Conclusions

Microwave-assisted synthesis successfully enabled the preparation of Al<sup>3+</sup>-modified P.O hybrid materials. Characterization analyses confirmed the incorporation of aluminum species and the modification of the fiber surface. The adsorption performance toward MO improved with increasing Al<sup>3+</sup> loading, reaching a maximum removal efficiency of 64.89% for P.O–4% Al<sup>3+</sup>. These results demonstrate that microwave-assisted Al<sup>3+</sup> functionalization is an efficient and eco-friendly approach for developing sustainable biosorbents for wastewater treatment.

### Acknowledgements

The authors thank LABEM (University of Sousse, Tunisia) for financial support, the GTS Research Group (Universitat Autònoma de Barcelona, Spain) for hosting us during the RAWMINA project, the Department of Mechanical and Nuclear Engineering, Khalifa University, Abu Dhabi, United Arab Emirates, and the NANOMISENE Laboratory (CRMN, University of Sousse, Tunisia) for their valuable support.



LinkedIn