STM JOURNALS

Journal of
Polymer & Composites

ISSN: 2321-2810 (Online)
ISSN: 2321-8525 (Print)
Volume 14, Issue 4, 2026
DOI (Journal): 10.37591/JoPC

https://journals.stmjournals.com/jopc

Research JoPC

Heavy Metal Removal from Textile Industrial Effluent using Banana Exocarp Derived Cellulose Acetate Graphene Nanocomposite Membranes

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Abstract

The disposal of textile effluent is responsible for serious human health concerns and above all environmental concerns to all kind of species. This indicates that there is immediate and urgent need of emerging, efficient with sustainable recycling technologies. In this paper, banana exocarp as agriculture-based waste, in terms of substrate was utilised as major raw material. The extracted cellulose from the substrate with homogeneous acetylation process, transformed into cellulose acetate (CA). Phase inversion method was used to prepare dense CA membranes. Thereafter graphene nanoparticles were incorporated in CA, which improvise affinity towards heavy metal ions, stability to dispersion and even membrane hydrophilicity. The experimental and analytical methods such as FESEM with EDS, XRD, FTIR and Contact Angle used compressive characterisation of fabricated membrane. The observations from all this analysis evaluated the structural, thermal and surface properties of casted membrane. This designed and formulated nanocomposite membrane were applied for recycling of textile effluent instead conventional process. The efficiency and performance of nano filtration was assessed for Ni^{2+}, Fe^{2+}, Zn^{2+} and Cu^{2+} ions. The enhanced membrane selectivity added advantage through adsorption sieving mechanisms and surface complexation with metal ions. In comparison with conventional CA membranes, the graphene nanocomposite CA membranes confirmed enhancement in removal efficiency of heavy metals from wastewater, achieving rejection rates of up to approximately 93% for Ni^{2+} and Fe^{2+}. The combined effect of CA and Graphene attributed to the improved version of Cellulose Acetate Graphene Nanocomposite Membrane. Overall, the findings indicate that cellulose acetate membranes derived from banana exocarp and modified with graphene offered a sustainable and effective solution as emerging treatment technology. In addition, these membranes exhibited superior antifouling characteristics and maintained stable permeate flux during operation.

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Received Date: April 23, 2026
Accepted Date: May 29, 2026
Published Date: June 09, 2026

Citation: Pragati More, Ashok More, Ashwini Patil, Avinash Pawar. Heavy Metal Removal from Textile Industrial Effluent using Banana Exocarp Derived Cellulose Acetate Graphene Nanocomposite Membranes. Journal of Polymer & Composites. 2026; 14(4): 51–62p.

Keywords: Cellulose acetate, banana exocarp, cellulose acetate graphene nanocomposite membrane, heavy metal removal, textile effluent

INTRODUCTION

Textile industries are among the major contributors to industrial water pollution due to the discharge of large volumes of wastewater containing dyes, salts, surfactants and toxic heavy metals [1, 2]. Heavy metals such as nickel (Ni^{2+}), iron (Fe^{2+}), zinc (Zn^{2+}) and copper (Cu^{2+}) are of particular concern owing to their non biodegradable nature, environmental persistence and tendency to

bioaccumulate in living organisms [3, 4]. Even at low concentrations, these metals pose significant risks to human health and aquatic ecosystems, including organ toxicity, neurological disorders and disruption of biological functions. Therefore, the development of efficient and sustainable technologies for heavy metal removal from textile effluents has become a critical research priority. Conventional wastewater treatment methods such as chemical precipitation, coagulation with flocculation, ion exchange and adsorption, have been widely employed for heavy metal removal. However, these approaches are often associated with limitations including high chemical consumption, generation of secondary sludge, operational complexity and reduced efficiency at low metal concentrations [5–7]. In this context, membrane based separation technologies have gained considerable attention as promising alternatives due to their high removal efficiency, compact system design and reduced environmental impact [8–10]. Among various polymeric membrane materials, cellulose acetate (CA) has emerged as a favourable candidate because of its biodegradability, low toxicity, cost effectiveness and excellent film forming properties [11, 12].

Recent research has increasingly focused on the utilization of agricultural waste as a sustainable source of cellulose, aligning with circular economy principles and waste valorization strategies [13, 14]. Banana exocarp is an abundant agro waste, offers a viable and eco friendly raw material for cellulose extraction. The conversion of such biomass into cellulose acetate not only minimizes environmental burden but also provides a cost-effective approach to membrane fabrication. Nevertheless, pristine CA membranes often exhibit inherent limitations such as moderate selectivity, susceptibility to fouling and limited affinity toward dissolved heavy metal ions, which restrict their performance in complex industrial wastewater systems [10, 15]

To address these challenges, the incorporation of nanomaterials into polymeric membranes has emerged as an effective strategy to enhance membrane performance. Graphene nanoparticles in particular, have attracted significant interest due to their high specific surface area, exceptional mechanical strength and strong interaction with metal ions. The integration of graphene into CA matrices improves membrane hydrophilicity, enhances dispersion stability and facilitates adsorption assisted separation mechanisms. Similar enhancements in membrane performance have also been reported with nanomaterial incorporation, including improved hydrophilicity, antifouling and metal ion rejection efficiency [16–18]. In this context, the present study focuses on the development of cellulose acetate graphene nanocomposite membranes derived from banana exocarp for the efficient removal of heavy metals from textile industrial effluent. The membranes were fabricated using the phase inversion technique and characterized using advanced analytical methods to evaluate their structural, morphological and surface properties. Their performance was assessed for the removal of Ni^{2+}, Fe^{2+}, Zn^{2+}, Cu^{2+} ions, with particular emphasis on selectivity, antifouling characteristics and permeate flux stability.

Overall, this study aims to develop a sustainable, cost-effective and high performance membrane based solution for wastewater treatment by integrating agricultural waste valorization with advanced nanocomposite technology. The findings are expected to contribute to the advancement of eco friendly membrane materials and support practical applications in textile effluent treatment [19] and water reuse systems.

MATERIALS AND METHODS

Materials

The materials employed in this study primarily consisted of agricultural biomass, chemical reagents, solvents, and nanomaterials essential for membrane fabrication and performance evaluation. Banana exocarp, an abundant agro waste rich in cellulose (approximately 35 – 45%), was utilized as the primary raw material for cellulose extraction, supporting sustainable resource utilization and circular economy principles [14] Analytical grade chemicals such as potassium hydroxide, sodium chlorite, hydrogen peroxide, glacial acetic acid, acetic anhydride and sulfuric acid were used for biomass pretreatment,

delignification, bleaching and acetylation processes to synthesize cellulose acetate (CA) [11, 13]. Organic solvents including acetone and N-methyl-2-pyrrolidone (NMP) were employed for membrane casting via the phase inversion technique [6, 8]. Graphene nanoparticles (GNP) were incorporated as nanofillers to enhance mechanical strength, hydrophilicity and adsorption affinity toward heavy metal ions [9]. The textile industrial effluent used for membrane performance evaluation was collected from a local processing unit and analyzed using standard procedures [5, 6]. All chemicals used were of analytical reagent (AR) grade mostly 'Aldrich' to ensure reproducibility and accuracy in experimental outcomes.

Methods

Extraction of Cellulose From Banana Exocarp

Cellulose was extracted from banana exocarp, an agricultural biomass rich in lignocellulosic components such as cellulose, hemicellulose, lignin, pectin and waxes. The extraction involved multistep chemical treatments including pretreatment, delignification and bleaching to obtain purified cellulose. Initially, banana exocarp was washed, cut into small pieces and treated with potassium metabisulfite to prevent oxidation and microbial degradation. Dewaxing was carried out using ethanol reflux, followed by drying at 60°C to remove moisture and preserve structural integrity. The dried biomass was pulverized to increase surface area for efficient chemical interaction. Delignification was performed using potassium hydroxide (KOH) under microwave assisted conditions [13, 14]. Microwave irradiation provided rapid volumetric heating, reducing reaction time and chemical consumption while improving lignin removal efficiency. Subsequently, bleaching was carried out using hydrogen peroxide under alkaline conditions to remove residual lignin and chromophores, yielding highly purified cellulose. Cellulose is isolated using microwave radiation at lower power 600W for repeated small cycles coupled with mechanical pretreatment, and subsequently converted to its derivative cellulose acetate.

Synthesis of Cellulose Acetate

Cellulose acetate (CA) was synthesized through an acetylation reaction, where hydroxyl groups (–OH) of cellulose were replaced by acetyl groups ($–OCOCH_3$), enhancing solubility, hydrophobicity and film forming properties [11, 12]. The extracted cellulose was first swollen in glacial acetic acid to facilitate reagent penetration. A catalytic mixture of sulfuric acid was added, followed by controlled addition of acetic anhydride as an acetylating agent. The reaction was maintained at 50°C for 1 hour, leading to dissolution and increased viscosity of the mixture. The reaction product was precipitated in cold distilled water, neutralized using sodium carbonate and washed to remove impurities. Finally, the cellulose acetate was dried to obtain a white polymer suitable for membrane fabrication. The degree of substitution governed the physicochemical properties of CA and its suitability for membrane applications [11].

Preparation of Graphene Nanoparticles (GNP)

Graphene nanoparticles (GNP) were synthesized using a combination of ultrasonication and microwave irradiation techniques [9, 16] Graphite was dispersed in dimethyl sulfoxide (DMSO) and subjected to ultrasonication to exfoliate graphene layers. The suspension was then irradiated in a microwave reactor at elevated temperature and controlled power, promoting efficient exfoliation and formation of graphene nanoparticles. The mixture was centrifuged to remove unexfoliated particles and filtered through a nylon membrane. The obtained product was dried at 80°C to yield graphene nanoparticles. Due to their hydrophobic nature and tendency to agglomerate, dispersion of GNP was achieved using ultrasonication in a solvent mixture of acetone and NMP. This ensured uniform distribution and stability of nanoparticles for subsequent membrane fabrication.

Incorporation of Graphene into Cellulose Acetate

Graphene nanoparticles were incorporated into the cellulose acetate matrix to form a nanocomposite. The CA polymer was dissolved in an acetone/NMP solvent system to form a homogeneous casting

solution [12]. Dispersed graphene nanoparticles were added to the solution and subjected to ultrasonication to ensure uniform dispersion [16]. The presence of graphene improved mechanical strength, thermal stability and adsorption affinity of the membrane, while also influencing pore structure and hydrophilicity [18]. Uniform dispersion was critical to avoid agglomeration, which could otherwise block membrane pores and reduce performance.

Formulation of Cellulose Acetate Graphene Nanocomposite Membrane

Membranes were formulated using the phase inversion technique, a widely used method for producing porous polymeric membranes [8, 12]. The prepared polymer nanoparticle solution was cast onto a glass plate using a controlled casting knife. The cast film was immediately immersed in a nonsolvent (distilled water) bath, inducing phase separation due to solvent–nonsolvent exchange. This resulted in the formation of a dense polymer matrix with porous structure suitable for nanofiltration [8]. Posttreatment involved immersion in distilled water for 24 hours to remove residual solvents. The resulting membrane exhibited improved permeability, selectivity and antifouling properties due to the incorporation of graphene nanoparticles [10].

Characterization

Characterisation of cellulose, cellulose acetate (CA) and cellulose acetate – graphene nanoparticle (CA – GNP) nanocomposite membranes was essential to evaluate structural, chemical, morphological, thermal and surface properties. These analyses provided insights into the transformation of biomass into functional membrane material and confirmed the successful incorporation of graphene nanoparticles within the polymer matrix [16].

Fourier Transform Infrared Spectroscopy (FTIR) analysis was performed to identify functional groups and confirm chemical transformations during cellulose extraction and acetylation [11]. The spectra were recorded in the range of 4000 – 500 cm^{-1}. The presence of hydroxyl (–OH) groups in cellulose confirmed its hydrophilic nature. After acetylation, characteristic peaks corresponding to carbonyl (C=O) stretching of ester groups confirmed successful formation of cellulose acetate [12]. In CA – GNP membranes, slight peak shifts and intensity variations indicated interactions between graphene nanoparticles and polymer chains. FTIR results confirmed the chemical modification from cellulose to cellulose acetate and the successful incorporation of graphene within the matrix.

X-Ray Diffraction (XRD) analysis was conducted to determine crystallinity and structural changes [13]. Native cellulose exhibited semicrystalline structure. After acetylation, crystallinity decreased due to disruption of hydrogen bonding. Incorporation of graphene slightly improved structural ordering due to its layered crystalline nature

The crystallinity index (CI) was calculated using;

$$\mathrm{CI}(\%) = \frac{\mathrm{I}_{200} - \mathrm{I}_{\mathrm{am}}}{\mathrm{I}_{200}} \times 100$$

Where: I_{200}= crystalline peak intensity, I_{am}= amorphous intensity

This analysis confirmed structural modification and enhanced crystallinity in nanocomposite membranes.

Field Emission Scanning Electron Microscopy (FESEM) was used to examine surface morphology and pore structure [15]. Pure CA membranes exhibited relatively smooth surfaces with limited porosity. CA – GNP membranes showed improved pore distribution and rougher surface morphology, indicating enhanced surface area. Graphene nanoparticles were observed as uniformly dispersed nanofillers. Energy Dispersive X-ray Spectroscopy (EDS) confirmed elemental composition and validated the presence of carbon rich graphene within the membrane matrix [15]. These results indicated improved

structural integrity and pore architecture due to graphene incorporation.

Thermogravimetric Analysis (TGA) was performed to evaluate thermal stability [18]. CA membranes showed moderate thermal degradation. CA – GNP membranes exhibited improved thermal stability due to graphene reinforcement. Graphene nanoparticles acted as thermal barriers, delaying decomposition and improving membrane durability under operational conditions.

Contact angle analysis was carried out to determine surface hydrophilicity [10]. Pure CA membranes showed moderate hydrophilicity. CA – GNP membranes exhibited reduced contact angle values, indicating improved wettability. Lower contact angle enhanced water permeability and reduced fouling tendencies, which is critical for wastewater treatment applications [20].

- *UV:* Visible spectroscopy was used to evaluate dye concentration and membrane rejection efficiency. Absorbance (200–800nm) were correlated with concentration using Beer–Lambert law [5]. Reduction in absorbance after filtration confirmed effective pollutant removal.
- *The performance of CA:* GNP nanocomposite membranes was evaluated for the removal of heavy metals from effluent under nanofiltration conditions. Heavy metal concentrations were quantified using Atomic Absorption Spectroscopy (AAS), a standard technique for trace metal analysis [6]. Metal ions were analyzed before and after filtration. This allowed accurate determination of removal efficiency.

RESULTS AND DISCUSSION

Structural and Chemical Analysis of Cellulose and Cellulose Acetate

The successful isolation of cellulose via microwave assisted extraction was confirmed through FTIR and XRD analyses. The FTIR spectrum as shown in Figure 1 exhibited characteristic peaks corresponding to hydroxyl (–OH), aliphatic C – H and β - glycosidic linkages as wavenumbers mentioned in Table 1, confirming the polysaccharide structure and removal of impurities.

Table 1. FTIR analysis of cellulose.

S.N.	Wavenumber (cm^{-1})	Functional group
1	3444	O – H stretching
2	2927	C – H stretching
3	1743	C = O stretching
4	1083	C – O stretching
5	906	B - glycosidic linkage

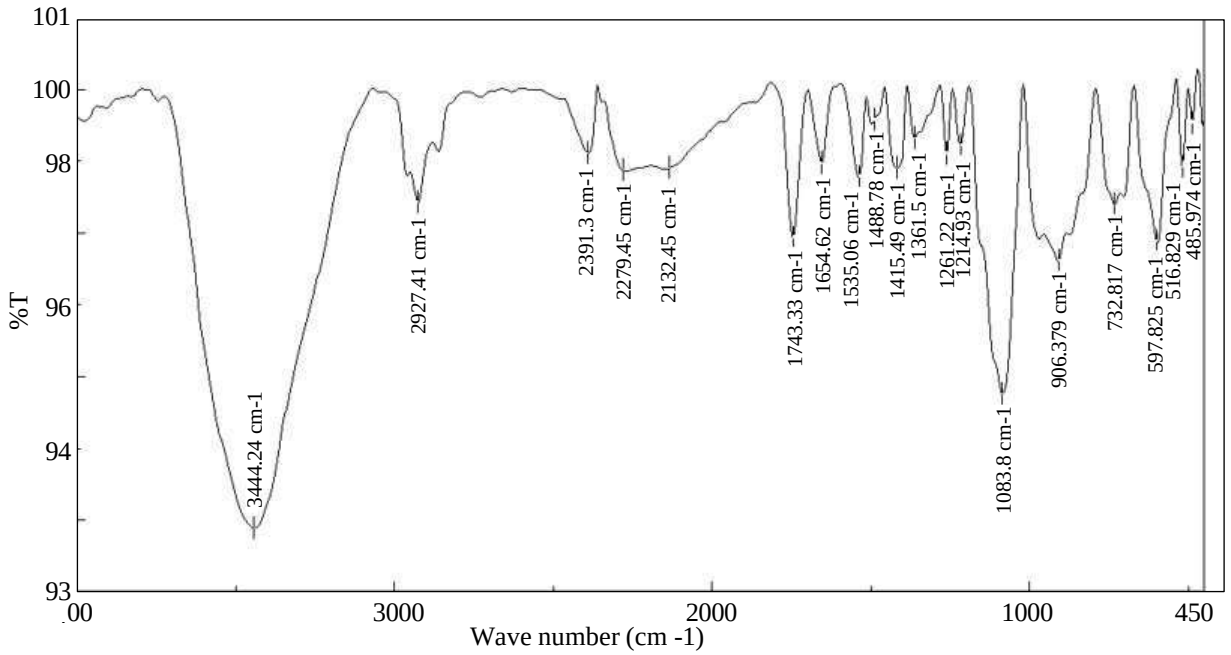

Figure 1. FTIR spectrum of pure cellulose. *(Testing Lab: PTL, Poona College of Pharmacy, Pune)*

XRD results shown in Figure 2 having strong diffraction at 2θ = 22.6°, confirming cellulose I crystalline phase with a crystallinity index of 82.5%, indicating improved structural order suitable for membrane fabrication.

Acetylation of cellulose to cellulose acetate (CA) was verified by the appearance of a strong carbonyl peak at 1756 cm^{-1} and reduction in hydroxyl intensity refer Figure 3, confirming substitution reactions.

XRD analysis as shown in Figure 4 revealed reduced crystallinity due to disruption of hydrogen bonding, leading to a more amorphous and processable polymer matrix.

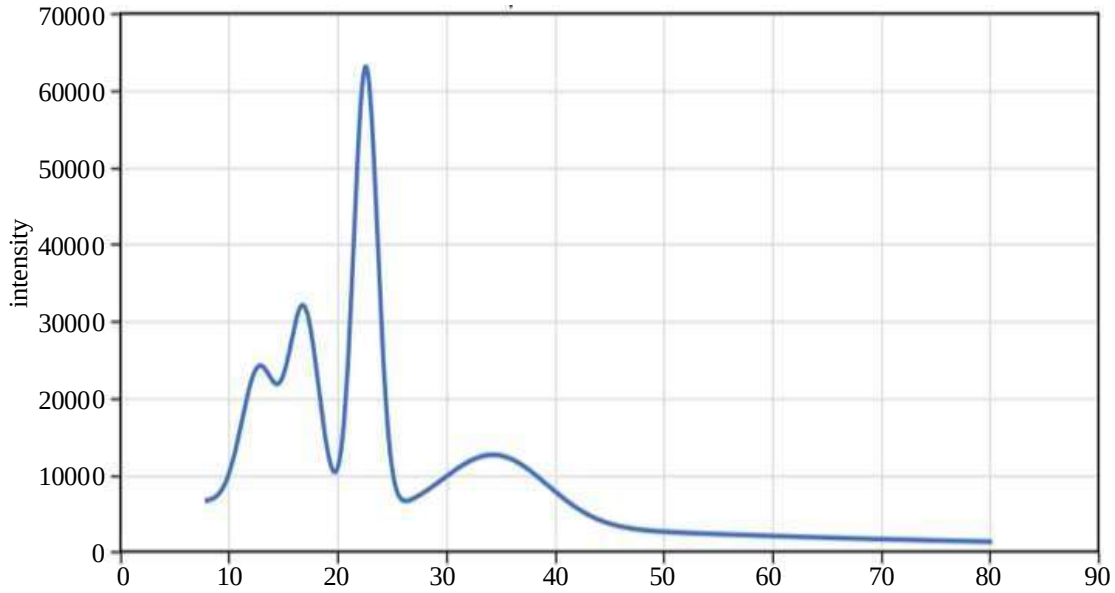

Figure 2. X-Ray diffraction spectrum of cellulose.
(Testing Lab: YCIS, Yashwantrao Chavan College, Satara)

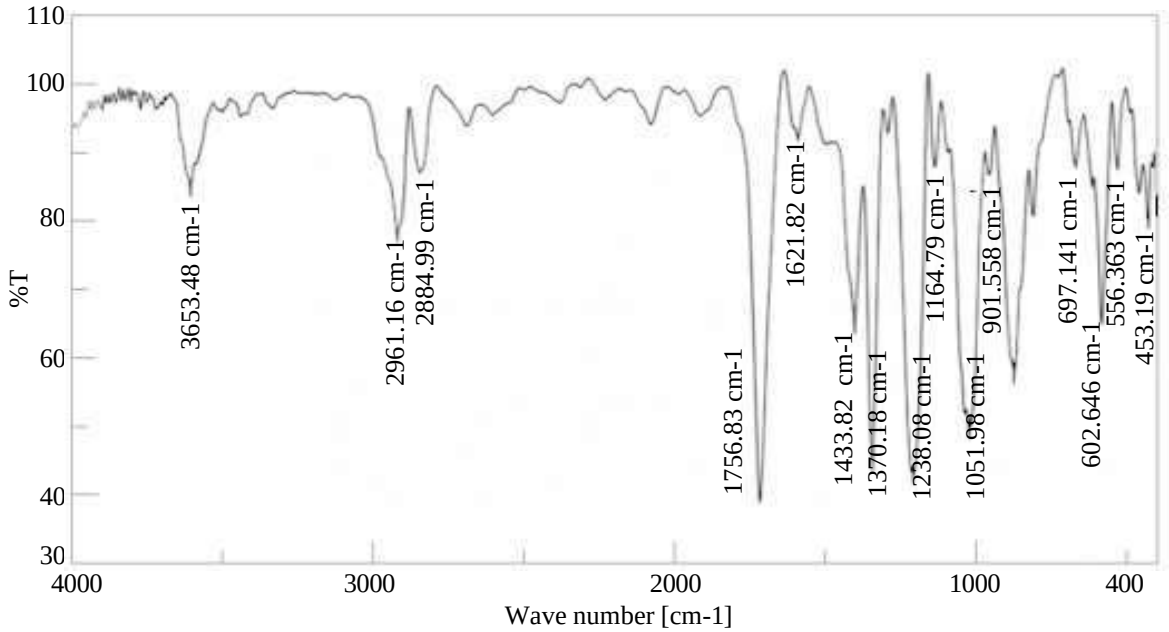

Figure 3. FTIR spectrum of cellulose acetate.
(Testing Lab: PTL, Poona College of Pharmacy, Pune)

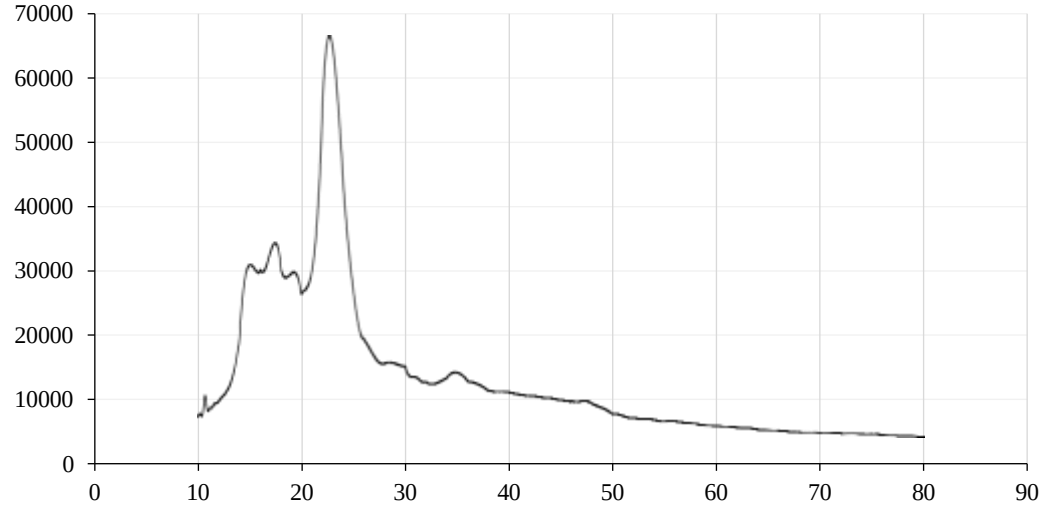

Figure 4. X Ray diffraction spectrum of cellulose acetate.
(Testing Lab: ATL, Yashwantrao Mohite College of Arts, Science and Commerce, Pune)

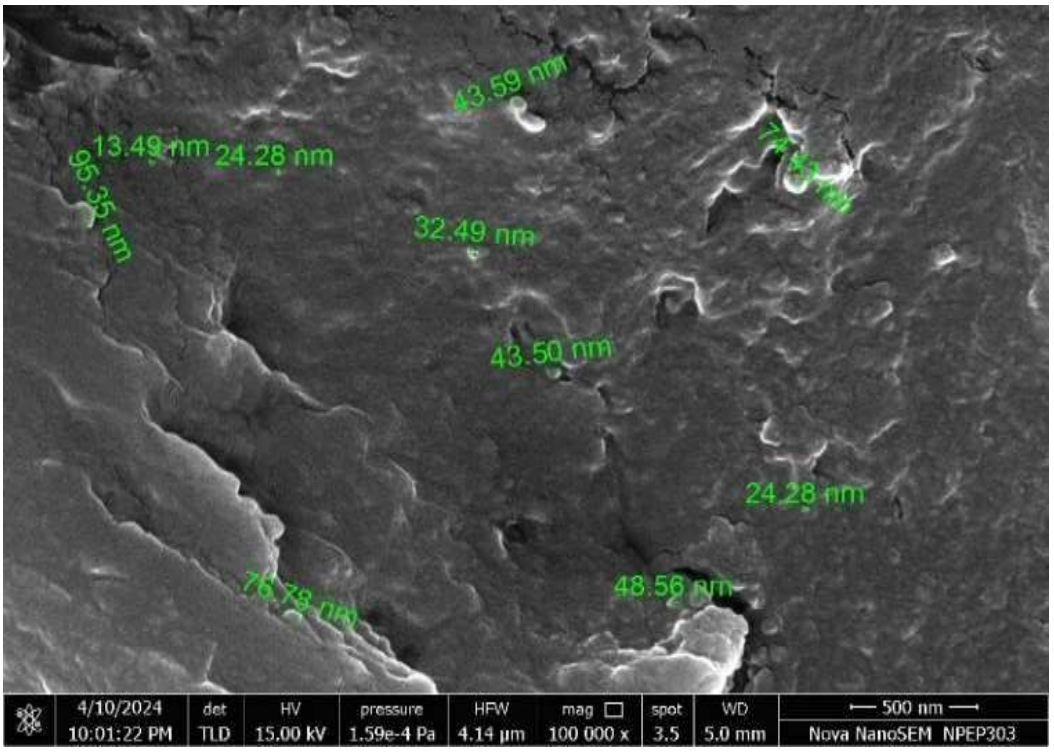

Figure 5. FESEM of cellulose acetate membrane.
(Testing Lab: CIF, Savitribai Phule Pune University, Pune)

Morphology and Properties of Cellulose Acetate Membrane

FESEM image as below in Figure 5 showed a smooth membrane surface with nanoscale pores (14 – 48 nm), indicating suitability for nanofiltration. The membrane exhibited a contact angle of 75.65°, confirming moderate hydrophilicity.

Thermogravimetric analysis showed in Figure 6 having two stage degradation, with major decomposition at 231°C, typical of CA polymers. Mechanical testing indicated tensile strength of 32 MPa, demonstrating balanced flexibility and strength.

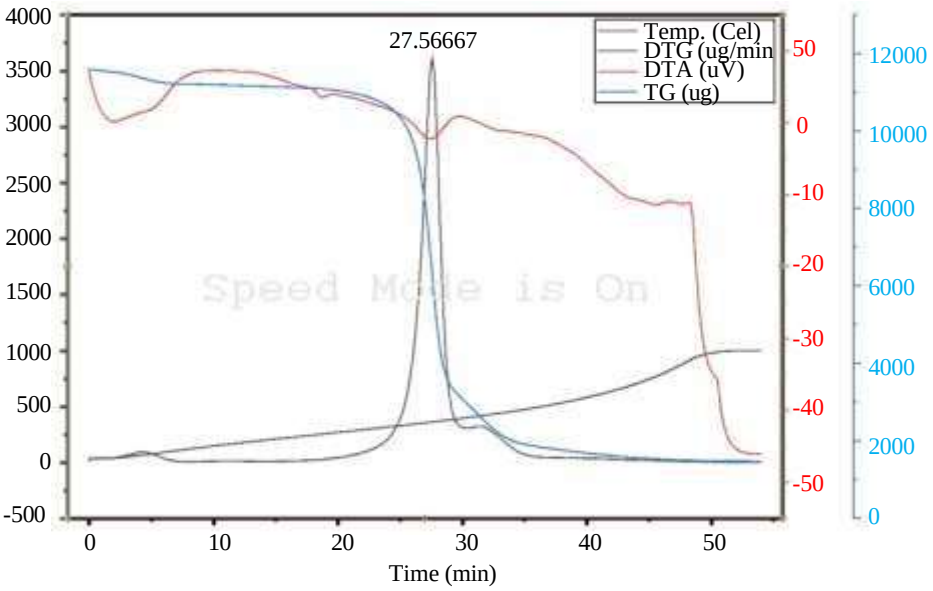

Figure 6. TGA curves of cellulose acetate membrane.
(Testing Lab: CL, Sadguru Gadge Maharaj College, Karad)

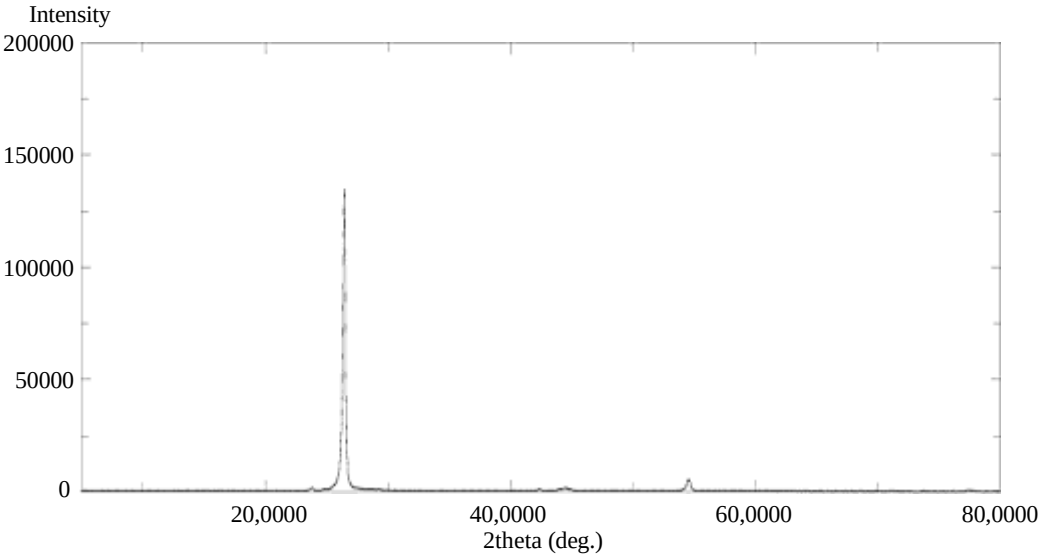

Figure 7. X-Ray diffraction spectrum of graphene.
(Testing Lab: AIF, Savitribai Phule Pune University, Pune)

Characterization of Graphene Nanoparticles (GNP)

Graphene synthesized via microwave irradiation and ultrasonication exhibited characteristic FTIR peaks for sp^2 carbon (1682 cm^{-1}) and minimal oxygen functionalities, indicating successful exfoliation. XRD peaks at 26.72° confirmed the (002) plane of graphene as indicated in Figure 7.

UV – Visible spectra shown in Figure 8, indicated absorption peaks at 223–273 nm, corresponding to π–π^* transitions, confirming conjugated carbon structures. FESEM images revealed thin, layered morphology [21] with minimal agglomeration.

Characterization of CA – GNP Nanocomposite Membrane

Chemical Interaction by FTIR spectra shown in Figure 9 as below, the emergence of C = C vibrations (~1654 cm^{-1}), confirming graphene incorporation. Broadening of –OH peaks indicated disruption of

hydrogen bonding and formation of new interaction sites.

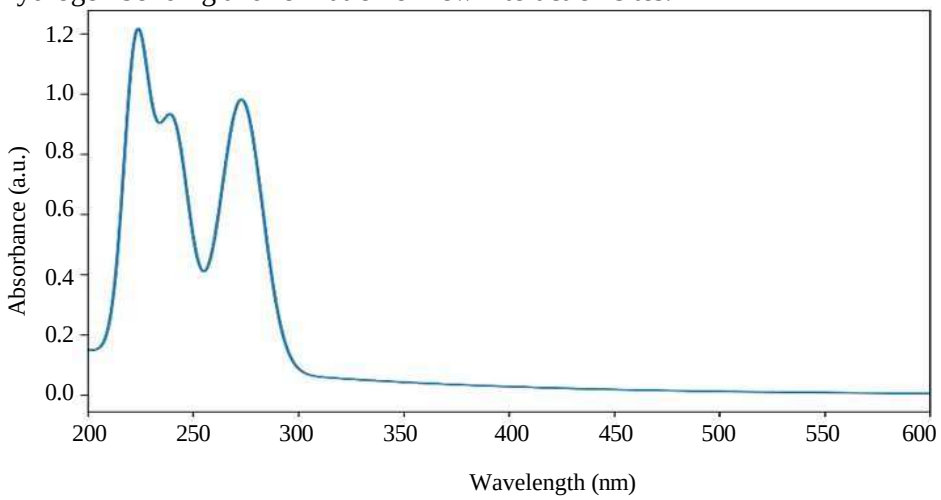

Figure 8. UV - Visible spectrum of GNP.
(Testing Lab: ATL, Yashwantrao Mohite College of Arts, Science and Commerce, Pune)

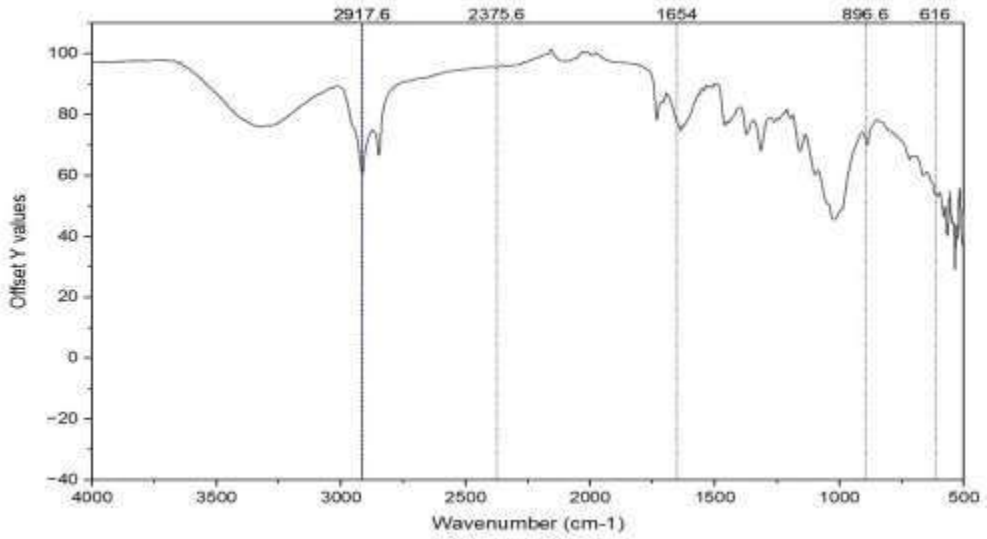

Figure 9. FTIR spectrum of CA – GNP Nanocomposite Membrane.
(Testing Lab: PTL, Sir Parashurambhau College, Pune)

Structural and Morphological Changes by XRD refer Figure 10, displayed broadened graphene peaks, indicating few layer graphene dispersion. FESEM (Figure 11) revealed increased surface roughness and layered morphology due to graphene incorporation. Thermal stability improved significantly, with degradation onset shifting to ~314°C. Tensile strength increased to 45 MPa due to graphene reinforcement. Surface Wettability by Contact angle increased to 84.65°, indicating increased hydrophobicity due to graphene sheets, influencing adsorption and fouling behaviour.

CA – GNP Nanocomposite Membrane Performance Evaluation

The CA – GNP membrane exhibited significantly higher flux due to enhanced pore connectivity and water transport channels. Permeate Flux of Membranes for CA and CA – GNP was observed to be 34.85 and 97.87 L/m²·h respectively. Dye Removal Efficiency was observed to be 99.18% and 93.18 % for CA - GNP and CA respectively. The superior performance of CA – GNP membranes is attributed to strong π–π interactions between graphene and dye molecules, along with high surface area.

Significant reductions in BOD and COD shown in Table 2 demonstrate the effectiveness of

nanocomposite membranes in removing organic pollutants.

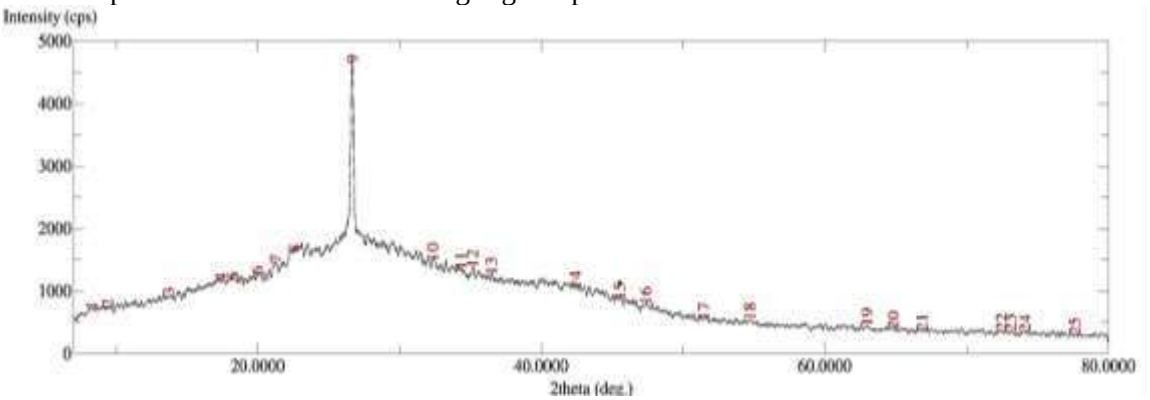

Figure 10. X Ray diffraction spectrum of CA – GNP nanocomposite.
(Testing Lab: YCIS, Yashwantrao Chavan College, Satara)

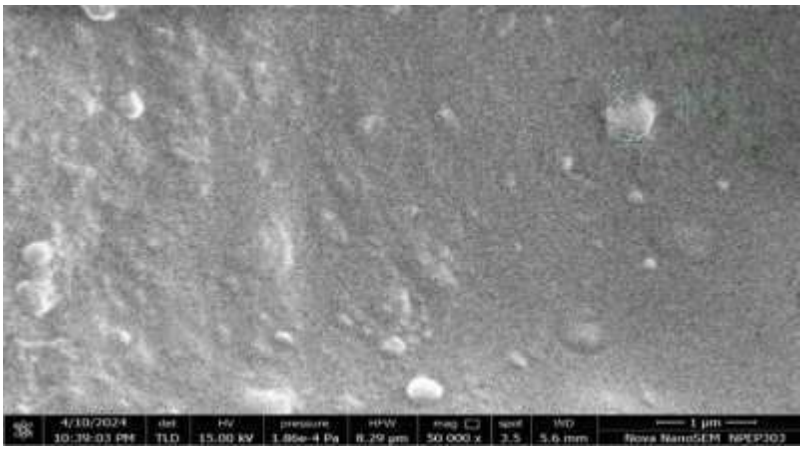

Figure 11. FESEM of CA – GNP nanocomposite membrane.
(Testing Lab: YCIS, Yashwantrao Chavan College, Satara)

Table 2. Effluent treatment performance.

Sample	pH	BOD (mg/L)	COD (mg/L)
Raw Effluent	8.5	865	1530
CA	7.3	690	1290
CA – GNP	6.9	410	625

The CA – GNP membrane demonstrated excellent heavy metal removal as shown in Table 3, due to adsorption mechanisms.

Table 3. Heavy metal removal efficiency (%).

Membrane	Fe	Cu	Ni	Zn
CA	72.71	78.22	82.29	74.99
CA – GNP	92.24	89.32	92.59	90.46

Table 4. Antifouling performance.

Membrane	FRR (%)	Rr (%)	Rir (%)
CA	54.57	36	45.42
CA – GNP	90.51	59.53	9.48

The CA - GNP membrane exhibited the highest antifouling performance due to enhanced

hydrophilicity and photocatalytic self cleaning properties, refer Table 4.

A strong correlation between structure and performance was observed;

Graphene incorporation → increased porosity, adsorption and mechanical strength

Nanocomposite architecture → enhanced flux, selectivity and stability

The synergistic interaction between polymer matrix and nanomaterials resulted in superior membrane performance for textile wastewater treatment.

CONCLUSIONS

The present paper has successfully demonstrated the development of a sustainable and high performance cellulose acetate – graphene nanoparticle (CA – GNP) nanocomposite membrane derived from banana exocarp for textile effluent treatment. The extraction process yielded highly crystalline cellulose with a crystallinity index of 82.5%, confirming structural suitability for membrane fabrication. Acetylation resulted in cellulose acetate with improved processability and membrane forming characteristics.

The fabricated CA membrane exhibited moderate performance with a permeate flux of 34.85 L/m²·h, tensile strength of 32 MPa, and contact angle of 75.65°, indicating balanced hydrophilicity. Incorporation of graphene significantly enhanced membrane properties. The CA – GNP membrane showed 2.8 times increase in flux (97.87 L/m²·h) and improved mechanical strength (45 MPa), confirming effective reinforcement.

In wastewater treatment performance, the CA – GNP membrane achieved superior dye removal efficiency of 99.18%, compared to 93.18% for pure CA. Significant reductions in organic pollutants were observed, with BOD reduced from 865 to 410 mg/L and COD from 1530 to 625 mg/L, demonstrating effective purification.

Heavy metal removal efficiency improved remarkably with graphene incorporation. The CA – GNP membrane achieved removal efficiencies of 92.24% (Fe), 89.32% (Cu), 92.59% (Ni) and 90.46% (Zn), compared to lower values for pure CA. Additionally, antifouling performance improved substantially, with flux recovery ratio (FRR) increasing from 54.57% (CA) to 90.51% (CA – GNP).

Overall, the synergistic interaction between cellulose acetate and graphene nanoparticles resulted in enhanced permeability, selectivity and stability. The developed CA – GNP nanocomposite membrane presents a cost effective, ecofriendly and efficient solution for advanced textile wastewater treatment and heavy metal removal.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the management and administration of Bharti Vidyapeeth Deemed to be University, Yashwantrao Mohite College of Art, Science and Commerce, Pune, India for providing the necessary institutional support, laboratory facilities and infrastructure to conduct this research. The authors also sincerely appreciate the continuous encouragement and moral support received from their families throughout the course of this work.

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