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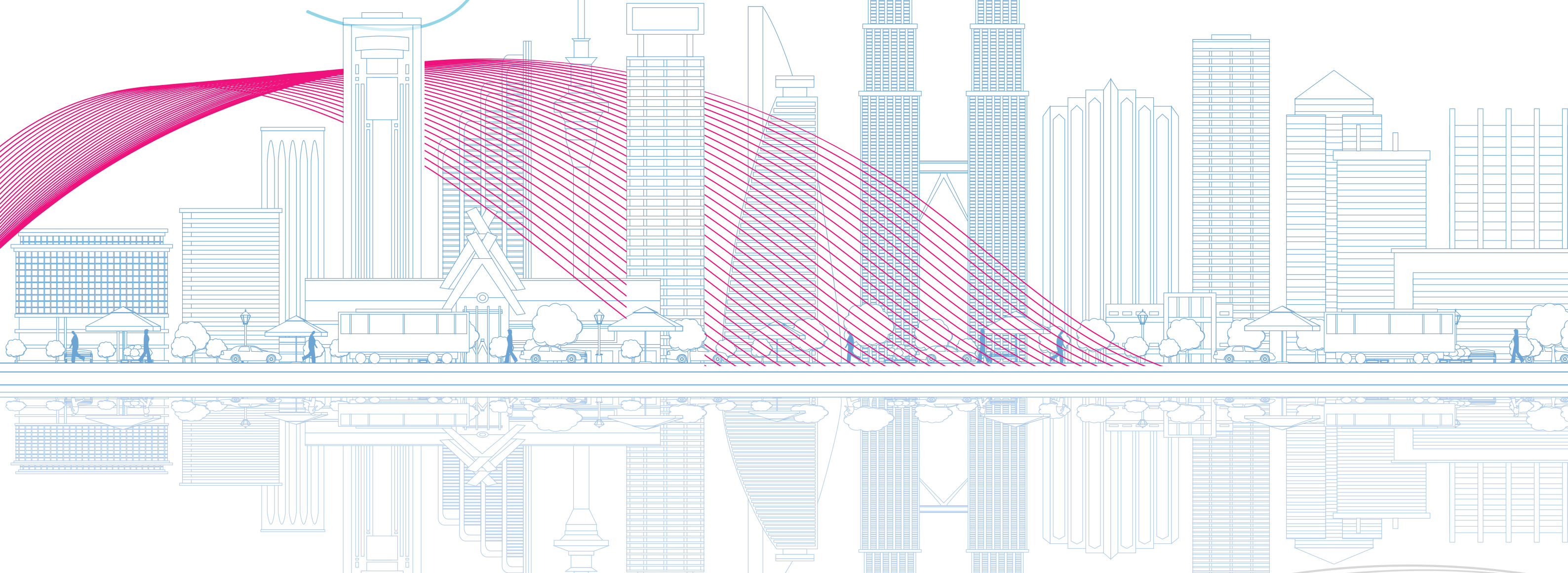
"Undergraduates' Digital Engagement Towards Global Ingenuity"

2nd Edition



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INNOVATION OF 3D-PRINTED WASTE-DERIVED GRAPHENE OXIDE FOR WATER TREATMENT VIA DIGITAL LIGHT PROCESSING

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Abstract
Water scarcity and contamination are pressing concerns that global society must address. Water supply and distribution infrastructure have barely improved despite urbanisation and community development. Broken pipes and heavy metal contamination from old, corroded pipelines may produce silt buildup, harbouring microbiological contaminants due to low chlorine levels. The technological limitations of conventional filtering technologies, which do not meet modern water treatment needs, may not efficiently eliminate all forms of waterborne pathogens. Therefore, this study focuses on advancing antibacterial filtration membranes for water filtration. This study presents artificial design structures integrating waste-derived graphene oxide (GO) into a membrane matrix via the additive manufacturing (AM) technique. This study aims to fill the membrane technology innovation gap by exploring new membrane fabrication alternatives involving designing and fabricating membranes using Digital Light Processing (DLP) to quickly print two-dimensional structural layers. Membrane performance will be optimised through spectroscopy, antibacterial analysis, water permeability, and selectivity analysis. This filtration membrane is unique in the realm of novel material composition. Interestingly, GO's hydrophilic oxygen functional group yields a strong affinity for water to other contaminants, allowing water to flow over its surface without experiencing friction. This facilitates the production of membranes with improved permeability, simultaneously addressing two critical aspects of water contamination by increasing the specific surface area and lowering membrane fouling. Considering the expansion of the global 3D printing market, the discoveries from this study could pave the way for a new manufacturing approach for producing filtration membranes utilising a diverse range of materials. This study aligns with the broader strategy to meet the Sustainable Development Goals (SDG), specifically SDG6, SDG9 and SDG11 and is situated within the framework of the Fourth Industrial Revolution (IR 4.0) with a particular focus on additive manufacturing of membrane designs for water.

Keywords: *Graphene Oxide, Water Treatment, Digital Light Processing*

1. INTRODUCTION

Problem statement

Approximately 1.2 billion people suffer from inadequate access to potable and clean drinking water, facilitating the spread of various contaminations. Heavy metals contamination in drinking water can have severe health implications, even at low concentrations. In contrast to organic pollutants, heavy metals such as lead (Pb), mercury (Hg), cadmium (Cd) and arsenic (As) are non-biodegradable and have a propensity to accumulate in living organisms. These heavy metal exposures and consumptions can cause neurological and developmental problems targeting the entire body system. Waterborne diseases have emerged as a pressing global concern as the need for safe drinking water escalates due to the challenges of overpopulation and urbanisation.

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Although much tap water is known to be safe to drink, the water that travels through the kilometre-long channel before reaching the residence is uncertain for direct usage. The sediment buildup, which harbours microbial pollutants due to low chlorine levels, might become the home for microbes. Some common microbial contaminants of concern in drinking water include bacteria (*Escherichia coli*, *Salmonella* spp.), protozoa (*Cryptosporidium* spp., *Giardia* spp.) and viruses (Allaq et al.,2023). Conventional water treatment methods, although somewhat effective, generally fail to entirely eradicate these microbial pollutants, particularly in areas with insufficient water treatment infrastructure. Disinfection has proven to be efficient against bacteria and viruses; however, it is ineffective against protozoa like *Cryptosporidium* spp. due to their strong resistance to chlorination. Hence, additional treatment methods such as filtration are necessary to address *Cryptosporidium* spp. successfully.

Membrane fouling is a critical problem that directly affects the performance of membranes. The pores on the surfaces of the membrane can become blocked by several types of contaminants, such as organic matter, microbes, and inorganic particles, which decrease the flow of permeate and result in a reduction in efficiency. Material limitations such as limited durability, chemical stability, and the delicate trade-off between selectivity (ability to remove contaminants) and permeability (flow rate) affect the overall effectiveness of treatment and the lifespan of the current conventional membrane. Therefore, the idea behind developing anti-fouling membranes is to reduce the frequency and intensity of fouling, while advancements in integrated treatment systems, which integrate membrane technology with other approaches like antibacterial characteristics, provide a more holistic approach to water treatment.

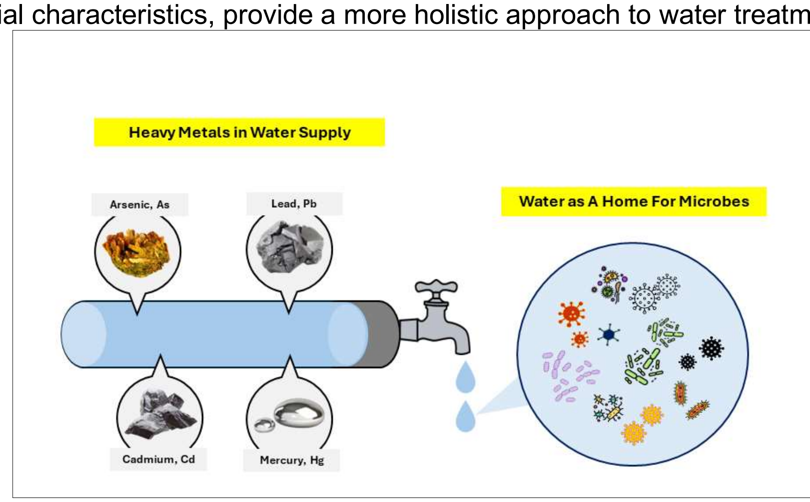

Figure 1 Problems associated with water supply worldwide

Literature Review

Over the years, multiple efforts have been made to implement diverse wastewater treatment technologies. The field of membrane technology has experienced substantial growth in recent decades, mostly due to its numerous advantages in water and wastewater treatment. The conventional membrane methods used for water purification and filtration include reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF), and microfiltration (MF). These processes are differentiated by the size of the membrane pores and the rejected solute **(Figure 2).** Additionally, existing conventional membranes have been enhanced to comply with current regulations for discharge or reuse. However, despite their efficacy, conventional membranes often suffer from fouling, limited permeability, and insufficient selectivity (Acarer, 2023).

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The primary mode of pathogen transmission to humans is through the consumption of water contaminated by humans and animals. Water contamination can occur through physical means (i.e., suspended particles), chemical means (i.e., heavy metals, dyes, pesticides, and pharmaceutical residues), or biological means (i.e., bacteria, fungi, viruses, and protozoa) (Punia et al.,2021). Contaminated drinking water caused by heavy metals released from different anthropogenic sources (mining, industrial, and agricultural activities) and waterborne pathogens like protozoan parasites (i.e., *Cryptosporidium* spp., *Giardia* spp.) and gram-negative bacteria (*Escherichia coli*, *Salmonella* spp.) in the water system poses many health risks and even causes serious diseases (Allaq et al., 2023).

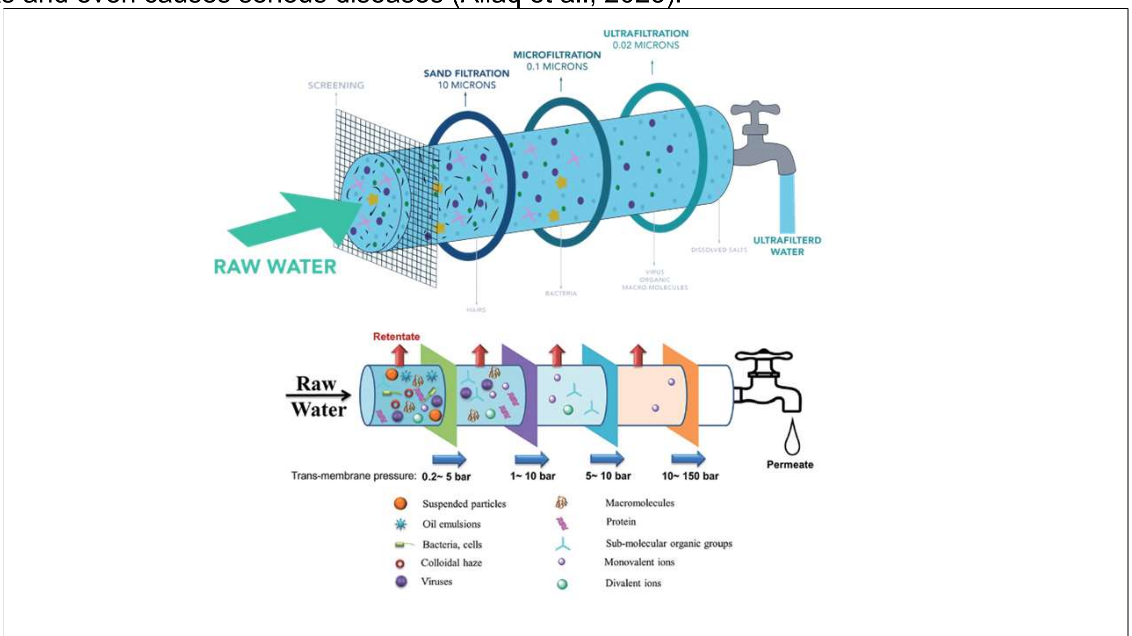

Figure 2 Conventional membrane processes in water purification filter (https://www.newater.com)

The adoption of membrane technologies for water purification has gained significant attention in ASEAN countries due to the pressing need for sustainable water management solutions. As an alternative to the conventional water treatment processes, membrane technologies have been developed to purify water from contaminated sources. Essentially, a membrane acts as a partition that separates two phases by selectively limiting the movement of components between them. These technologies offer great promise for applications in water purification, chemical separations, and various environmental and industrial applications, where membranes separate different gases, solids, and liquids (Barhoum et al., 2023).

With the discovery of nanomaterials, an incredible breakthrough has been made towards developing high-performance membranes. Recently, graphene oxide (GO), the most explored two-dimensional (2D) atom-thick layered nanomaterial (Geng et al., 2023), has been considered a promising candidate for the fabrication of new-generation membranes. Recent studies have demonstrated the effectiveness of GO-based membranes in removing a wide range of contaminants from water sources, including heavy metals, suspended solids, organic pollutants, and pathogens (Ahmed et al., 2021; Tiwary et al., 2024). Integrating polymer and GO into the same membrane composite structure offers various advantages in water purification. Adding GO to a pure polymer membrane improves the mechanical, thermal, and separation properties.

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Generally, the GO structure (Figure 3) contains oxygen-containing functional groups, such as carboxyl groups (-COOH), hydroxyl groups (-OH), and epoxy groups (-C-O-C), which contribute to the hydrophilicity and dispersity of GO in any aqueous solution (Iqbal et al., 2024). These groups can form hydrogen bonds with solvent molecules, resulting in the formation of a stable suspension rather than the formation of agglomerates (Tewari et al., 2024). Therefore, the feed water will not dissolve the polymer membrane during filtration. The strong interfacial bonding between graphene and the surrounding matrix material facilitates effective load transfer. When the membrane is subjected to mechanical stress, the load is efficiently transmitted from the weaker matrix to the more resilient graphene particles, thereby enhancing the strength and endurance of the membrane. The hydrophilic oxygen functional groups in GO yield a strong affinity for water over contaminants (Elzubair et al., 2024), allowing water to flow over its surface without experiencing friction. This facilitates the production of highly effective membranes with improved permeability and reduced membrane fouling.

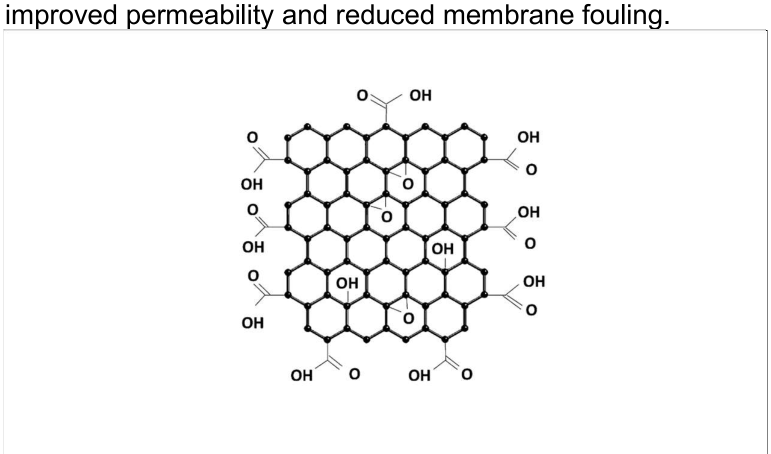

Figure 3 Chemical structure of graphene oxide

The high surface area and presence of functional groups in GO enable efficient adsorption and filtration of contaminants, opening new avenues for enhancing water filtration technologies with antibacterial capability (Geng et al., 2023; Elzubair et al., 2024). The antibacterial efficacy of GO stems from its capacity to mitigate waterborne diseases by combating bacteria and biofouling (Shahnaz & Hayder, 2023). GO exhibits tremendous antibacterial potential against various bacterial species, including Gram-positive and Gram-negative bacteria, achieved through various mechanisms (Figure 4), such as oxidative stress, edge-cutting, and disrupting the cell membrane via the cell entrapment effect (Xia et al., 2019; Omran & Baek, 2021).

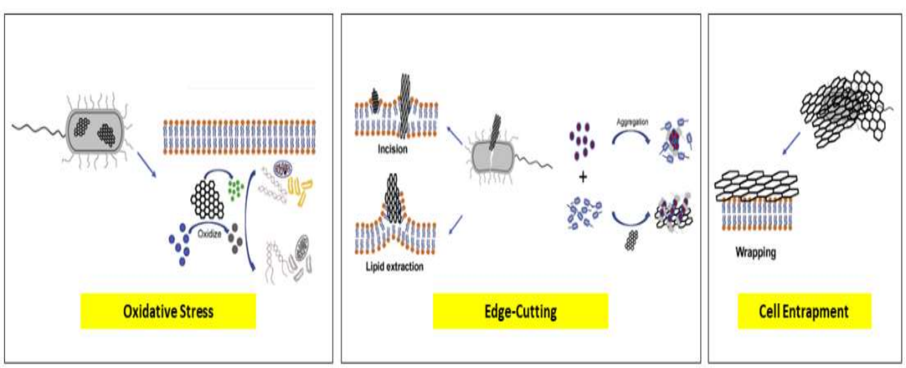

Figure 4 Established antibacterial mechanisms of graphene oxide (M.-Y. Xia et al., 2019)

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Additive manufacturing (AM), or 3D printing, is a rising manufacturing method that has shown notable progress across various industries. It uses computer-aided designs to precisely and directly deposit material layers to form a three-dimensional shape (Zhang et al., 2024; Rashid et al., 2021) from a digital file. AM has continuously developed and enhanced to utilise preexisting and newly developed materials (Rashid et al., 2020). Various additive manufacturing techniques such as stereolithography (SLA), fused deposition modelling (FDM), and selective laser sintering (SLS) offer innovative solutions for membrane fabrication (Koo et al., 2021). AM offers a transformative approach to membrane fabrication, enabling precise control over structural parameters and the ability to design membranes with tailored properties. A report (**Figure 5**) published by ThyssenKrupp estimated that AM would generate a hundredth billion worth of economic value in ASEAN by 2025 and 3 to 4 million additional jobs by 2030. This report provides a road map for increasing the penetration of additive manufacturing in the ASEAN manufacturing market.

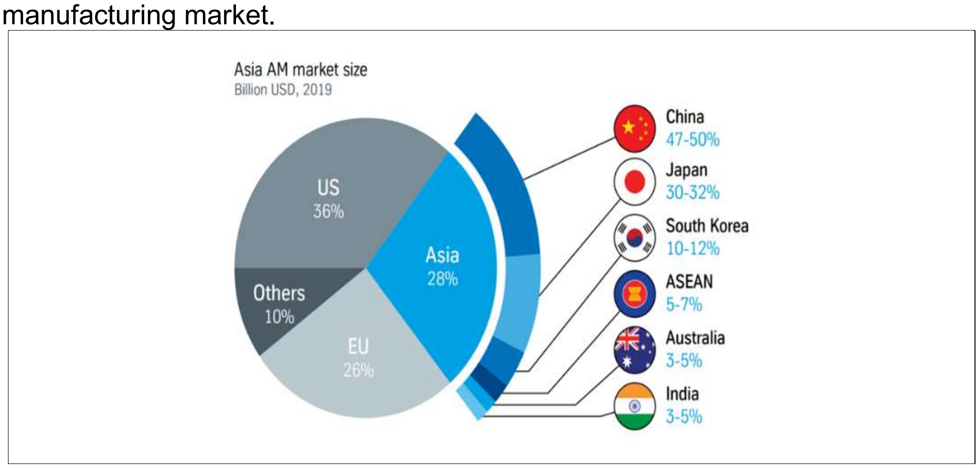

Figure 5 Additive Manufacturing Market Size in Asia

Emerging techniques like AM allow for the exact manipulation of membrane structure, surface properties, and distribution of pore sizes, resulting in membranes that can be customised to enhance performance and reduce fouling. Common materials used in AM for membrane fabrication include polymers, ceramics, and metals, selected based on their compatibility with AM processes and suitability for water filtration (Barman et al., 2023). Polyurethane acrylate (PUA) is a particular polymer membrane that has been recognized as having excellent mechanical properties, flexibility and durability. While PUA offers a few advantages as water filtration membranes, it also has some drawbacks such as hydrophobicity and fouling susceptibility (You et al., 2023).

The bonding between graphene and polymer matrix can effectively improve the structural performance of the composites, while the configuration of the composites can be controlled based on the design capabilities of 3D printing (You et al., 2023). Zhang *et al*. reported that three-dimensional graphene-based adsorbents prepared by AM technology generally perform better (Zhang et al., 2023). Despite the complexity of graphite flakes and structural defects posing challenges in understanding detailed oxidation mechanisms for graphene-based membranes, a similar observation by Valentin et al. (2019) showed that the hydrogels with GO addition prepared by light-directed 3D printing exhibit improved mechanical properties, excellent stability in high salt solutions, and superoleophobic characteristics. These membranes, typically in thin film composite form, have demonstrated improved solvent permeance in water treatment processes, serving as a valuable reference for potential use in organic solvent nanofiltration (Figure 6) (Nie et al., 2021).

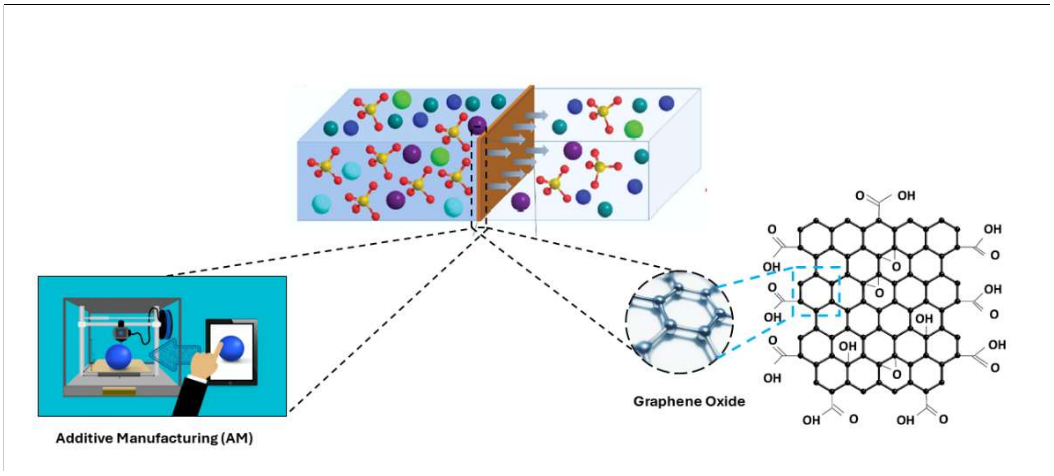

Figure 6 Schematic diagram for permeation through GO membrane

Integration of additive manufacturing and graphene-based materials for the fabrication of filtration membranes offer significant improvements in permeability and selectivity compared to conventional membranes. The ability to fine-tune pore structures and surface chemistry allows these membranes to achieve high water flux rates while maintaining excellent contaminant rejection rates. AM membranes can outperform conventional membranes by providing better flux and rejection performance, thus showing significant potential for advanced water filtration and purification applications (Tijing et al., 2020).

Objectives

- To determine the antibacterial properties of the 3D-printed GO/PUA membrane against waterborne pathogens (*E. coli* and *Salmonella* spp.) using the disc diffusion method.
- To evaluate the removal performance of Lead (Pb), Mercury (Hg), Arsenic (As), and Cadmium (Cd) of the 3D-printed membrane.
- To assess the performance of the 3D-printed membrane through water permeability, selectivity test, and mechanical analysis.

2. MATERIALS AND METHODS

Stage 1

Preparation of PUA/GO Blend Resin Compositions

GO powders, successfully synthesized using waste resources from our group and previously reported, are dissolved in the PUA resin, followed by a stirring process. All PUA/GO blend compositions are prepared using the polymer blending method with different GO weight percentages of 10, 15, 20, and 25 wt.%.

Stage 2

Characterization of PUA/GO Blend

Pure PUA and PUA/GO blend resin underwent viscosity analysis to determine their viscosity value and printability.

Stage 3

Three-Dimensional Model Design and Printing Process of PUA/GO Membrane

Initially, all the 3D-printed samples are designed using 3D-Printing software, and rendered designs are made viewable and readable for use in a 3D digital light processing (DLP) printer. The study prints the designed film samples using a 3D DLP printer. The homogenous PUA/GO liquid resin mixture is filled into a resin tray (vat).

The light source from the DLP projector, directed to the vat from the bottom, solidifies the resin in the desired pattern, and the printed layers are dragged upwards until completion. A triplicate of samples for each composition of the PUA/GO blend are printed in this study.

Stage 4, Stage 5 and Stage 6
Analysis & Characterization of 3D-Printed PUA/GO Membrane
All the 3D-printed PUA/GO samples underwent several characterizations, including surface morphology by (FESEM, elemental composition analysis by (FESEM-EDX), crystallinity by XRD, chemical functional groups by FTIR, mechanical analysis, and antibacterial efficiency analysis. The removal performance of lead (Pb), mercury (Hg), arsenic (As), and cadmium (Cd) of the 3D-printed membrane is evaluated using ICP-MS. The separation performance of fabricated 3D-printed PUA/GO membranes is assessed through water permeability and selectivity tests. Water permeability and selectivity analyses determine the suitability for specific separation processes. The antibacterial properties of the 3D-printed PUA/GO membrane are determined using the disc diffusion method against *E. coli* and *Salmonella* spp. The impregnated disc is then dipped into a PUA and PUA/GO blend resin and dried prior to being placed on the Petri dish containing bacteria. After 24 hours, the inhibition zone is measured to determine the antibacterial capability of the PUA and PUA/GO blend resin.

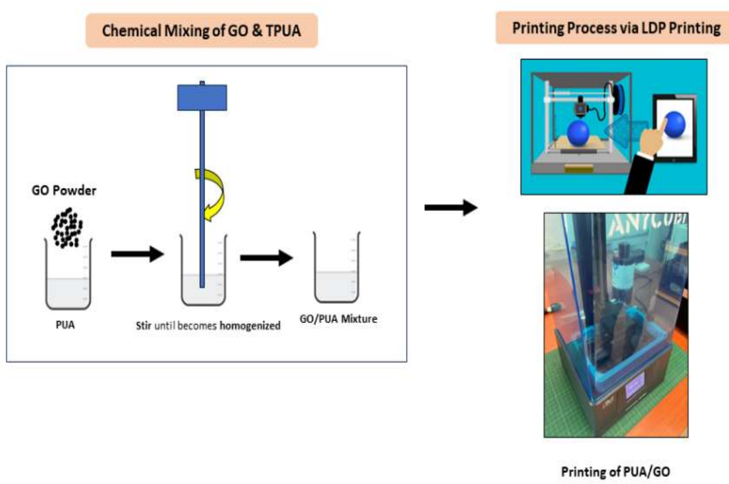

Figure 7 Schematic Illustration of overall methodology

3. RESULTS AND DISCUSSION

This study represents the first approach to developing a novel fabrication method for producing separation membranes made of antibacterial PUA/GO from waste-derived graphene, utilising additive manufacturing techniques. The capability to create customised membrane designs through AM showcases a potential approach for developing membranes with improved water permeability and selectivity performance. The ultimate goal is to facilitate the adoption of these advanced filtration membranes to improve the water quality through three key aspects:

- ICT in membrane fabrication and monitoring through the Internet of Things (IoT) enables continuous monitoring and control over various parameters to optimise membrane design and functionality. This integration helps ensure that the membranes produced meet the desired performance criteria of water treatment applications.
- The precision of additive manufacturing as an innovative engineering approach in synthesising and integrating waste-derived graphene oxide into advanced filtration membranes.

- The entrepreneurial approach for commercialisation involves promoting sustainable and marketable water treatment technologies, achieved by utilising low-cost starting waste materials and advanced manufacturing techniques that enhance efficiency and cost-effectiveness. The innovative nature of this project can lead to new business opportunities, aligning with the entrepreneurial spirit of innovating for tomorrow.

4. CONCLUSION

- The 3D graphene oxide membrane made from coconut shell waste has great promise for the water treatment industry.
- The membrane's mechanical, structural, and thermal characteristics have all been significantly enhanced with the addition of graphene oxide, leading to better filtration performance.
- The addition of GO enhances the strength of the membrane due to improvement in yield strength, and the membrane can be used at elevated temperatures since higher thermal degradation is obtained.
- In addition to addressing the problem of agricultural waste, this sustainable strategy provides an economical and environmentally beneficial method of purifying water.

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I am writing to express my gratitude to the team members, Zata and Hafiz, and Ts. Dr. Noor Asnida Asli for the unwavering support to the project titled "Innovation of 3D-Printed Graphene Oxide For Water Treatment Via Digital Light Processing." The project's primary goal is to develop an innovative fabrication method to produce high-performance 3D-printed GO/PUA membranes using waste-derived graphene and additive manufacturing techniques. This project presents a groundbreaking approach to addressing significant gaps in current water filtration membrane technologies. By leveraging waste-derived graphene oxide (GO) and combining it with polyurethane acrylate (PUA), this research aims to create a highly efficient and sustainable antibacterial filtration membrane. The use of additive manufacturing (AM) techniques allows for precise manipulation of membrane structure, optimizing performance, and reducing fouling, which is a common issue in traditional membranes.

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