

MICELLE-LADEN HYDROGEL TO REMOVE HYDROPHOBIC MICROPOLLUTANTS FROM WASTEWATER

AN HONOURS MINI PROJECT REPORT

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In

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DECLARATION

We hereby declare that the Mini Project report “**MICELLE- LADEN HYDROGEL TO REMOVE HYDROPHOBIC MICROPOLLUTANTS FROM WASTEWATER**”, submitted for partial fulfilment of the requirement award of honour degree of Bachelor of Technology of the APJ Abdul Kalam Technological University, Kerala is a bonafide mini project report done by us. The submission represents our idea in our own words and where ideas of others have been included we have adequately and accurately cited and referenced the original source. We also declare that we have adhered to ethics of academic honesty and integrity and have not misrepresented or fabricated any data or idea or fact in my submission. We understand that any violation of the above will cause disciplinary action by the institute and or the University and can also evoke penal action from the sources which have thus not been properly cited or from whom proper permission has not been obtained. This report has not been previously formed the basis for the award of any degree, diploma or similar title of any other University.

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CERTIFICATE

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PROJECT GUIDE

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ABSTRACT

Wastewater micropollutants such as pharmaceuticals, personal care products, pesticides, and industrial chemicals are emerging environmental and public health challenges as a result of their persistence and bioaccumulation effects on the aquatic environment and human health. The contaminants persist through almost all the treatment processes and thus present a tricky challenge. This challenge requires advanced treatment technologies and policies to curb the potential impact on water quality.

Hydrogel-based polymeric absorbents with immobilized micelles are presented in this work as a means of eliminating hydrophobic micropollutants from water. Synthesized acrylated surfactants self-assemble to form micelles with hydrophobic centres. Through free radical polymerization, the acrylate groups allow micelles to be incorporated into poly (ethylene glycol) diacrylate (PEGDA) hydrogels. The cross-linked hydrogels are characterized using NMR spectroscopy. To increase surface area and speed up uptake, droplet-based microfluidics is used to manufacture the hydrogels into microparticles. The immobilized micelles within these microparticles rapidly partition hydrophobic micropollutants, with mass transfer coefficients higher than those found for a commercially available activated carbon that is commonly used for water filtration. Lastly, a simple and sustainable process for recovering used absorbent is presented.

Keywords: Hydrogel, Micropollutants, Micelle, Hydrophobic.

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ABBREVIATION

PPCP- Pharmaceuticals and Personal Care Products

POP - Persistent Organic Pollutants

PCB - Polychlorinated Biphenyls

PFAS - Polyform Alkyl Substances

NSAID- Non-Steroidal Anti-Inflammatory Drug

CAFO - Concentrated Animal Feeding Operation

UV- Ultraviolet

FTIR - Fourier Transform Infrared Spectroscopy

HNMR - Higher Resolution Nuclear Magnetic Resonance

SEM- Scanning Electron Microscopy

PI - Photo Initiator

PEGDA- Poly Ethylene Glycol Diacrylate

pH-Potential of Hydrogen

NVP - N Vinyl Pyrrolidone

IR - Infrared

PVP - Poly Vinyl Pyrrolidone

PAA - Peracetic Acid

SLES - Sodium Laureth Sulphate

CHAPTER 1

INTRODUCTION

1.1 GENERAL BACKGROUND

Water is essential for human life and integral to sustainable economic development. The used water produced by industrial operations, everyday activities, and even rainfall runoff is known as wastewater. This includes the water used in factories, the water that drains from showers, toilet flushes, and laundry loads, as well as the rainwater that collects contaminants as it cascades over surfaces. Even though the majority of wastewater is just water, there are several toxins in it that must be removed before it can be properly released back into the environment or even used again for other reasons.

Macro and micropollutants are the two categories into which pollutants can be divided. Agricultural runoff containing phosphates and nitrates, bacteria such as coliforms, and other organic materials are examples of macro pollutants, but they are few in quantity and account for the majority of water pollution. On the other hand, there are many different types of micropollutants, and each one contributes very little to the overall mass of water pollution. Some examples of micropollutants are industrial organic solvents, lubricants and intermediates, industrial surfactants, detergents and disinfectants for the home, antibiotics and other pharmaceuticals, specific heavy metals, food additives and flavouring compounds, and nanomaterials (Devashish Gokhale et al.,2022).

1.1.1 MICROPOLLUTANTS

Wastewater micropollutants pose a serious threat to the ecosystem. These contaminants include a wide range of compounds, from industrial chemicals and pesticides to medications and

personal hygiene items. Micropollutants are invisible to traditional wastewater treatment systems because of their low concentrations. As a result, they remain in effluent and endanger human health and aquatic ecosystems. Their influence can be amplified by their capacity to bioaccumulate in organisms, which may result in endocrine system changes, abnormalities related to reproduction, and the growth of bacteria resistant to antibiotics. To efficiently remove micropollutants from wastewater, the study highlights the necessity for sophisticated treatment technologies such as activated carbon adsorption, membrane filtration, and advanced oxidation processes. Additionally, to minimize their release into the environment and protect the quality of the water for both present and future generations, strong monitoring programs and strict regulatory measures are necessary (Escher et al.,2018).

1.1.2 CLASSIFICATION OF MICROPOLLUTANTS

Contaminants known as micropollutants are present in water systems, frequently in tiny amounts, but they have the potential to have serious negative effects on the environment and human health. They come from a variety of places, including as industrial operations, medications, personal care items, and agricultural runoff. For management and mitigation measures to be effective, it is essential to comprehend how micropollutants are classified. Here is a grouping according to their chemical makeup and place of origin:

1. **Pharmaceuticals and Personal Care Products (PPCPs):** These include medications, hormones, fragrances, and other chemicals from personal care products. They enter wastewater primarily through human and veterinary use.
2. **Pesticides and Herbicides:** Chemicals used in agriculture to control pests, insects, and weeds. They enter water systems through runoff and leaching from agricultural fields.

3. **Industrial Chemicals:** This category encompasses a wide range of substances used in industrial processes, including solvents, heavy metals, flame retardants, and plasticizers. Industrial discharges and runoff contribute to their presence in water bodies.
4. **Persistent Organic Pollutants (POPs):** These are organic compounds resistant to environmental degradation. Examples include polychlorinated biphenyls (PCBs), dioxins, and certain pesticides. They persist in the environment and accumulate in organisms.
5. **Emerging Contaminants:** Newly identified pollutants that may not have been previously regulated or monitored. Examples include per- and polyfluoroalkyl substances (PFAS), pharmaceutical metabolites, and nanomaterials.
6. **Microplastics:** Small plastic particles (<5 mm) originating from various sources such as synthetic clothing, personal care products, and plastic debris breakdown. They pose threats to aquatic life and can enter the food chain.
7. **Metals:** Heavy metals such as lead, mercury, cadmium, and chromium, originate from industrial processes, mining activities, and urban runoff. They can bioaccumulate in organisms and cause adverse health effects.

Table 1.1 Sources of micropollutants in the aquatic environment (Luo et al. 2014)

Category	Important subclasses	Major Sources	
		Distinct	Nonexclusive
Pharmaceuticals	NSAIDs, lipid regulators, anticonvulsants, antibiotics, β -blockers, and stimulants	Domestic wastewater (from excretion) Hospital effluents Run-off from CAFOs and aquaculture	Sources that are not exclusive to individual categories include: Industrial wastewater (from product manufacturing discharges)
Personal care products	Fragrances, disinfectants, UV	Domestic wastewater (from	

	filters, and insect repellents	bathing, shaving, spraying, swimming, etc.)	Landfill leachate (from improper disposal of used, defective, or expired items)
Steroid hormones	Estrogens	Domestic wastewater (from excretion) Run-off from CAFOs and aquaculture	
Surfactants	Non-ionic surfactants	Domestic wastewater (from bathing, laundry, dishwashing, etc.) Industrial wastewater (from industrial cleaning discharges)	
Industrial chemicals	Plasticizers, fire retardants	Domestic wastewater (by leaching out of the material)	
Pesticides	Insecticides, herbicides and fungicides	Domestic wastewater (from improper cleaning, run-off from gardens, lawns roadways, etc.) Agricultural runoff	

To safeguard water quality and public health, it is crucial to create efficient monitoring, treatment, and regulation systems that consider the many sources and characteristics of micropollutants.

1.1.3 INTRODUCTION OF HYDROGEL FOR MICROPOLLUTANT REMOVAL

Over the past few decades, hydrogels have been extensively used in a wide range of significant application fields, such as artificial snow, coal dewatering, agriculture, food additives,

pharmaceuticals, and biomedical applications like wound dressing, tissue engineering, and regenerative medicine (Talaat et al, 2008). Hydrogels have been used for a long time in several of the aforementioned application areas, particularly in the biomedical field, although reports of their use in water treatment date back barely ten years. Hydrogels can be synthetic or natural. Natural hydrogels are formed by polymers such as proteins and polysaccharides. However, synthetic hydrogels are the most used hydrogels in all of the aforementioned domains because of their increased efficiency (Zhang et al., 2011).

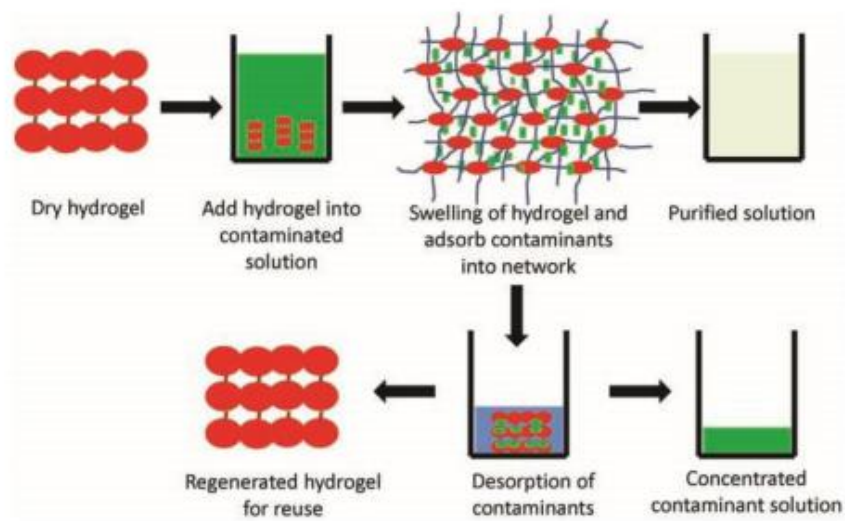


Figure 1.1 The way hydrogels act in water purification by contaminant adsorption (Lakshika Weerasundara et al., 2020).

Polymeric networks possessing strong water absorption and pollutant immobilization characteristics are the most promising hydrogels for water purification. These polymeric hydrogels are regarded as advanced hydrogels since they may exhibit various qualities in terms of gel volume, swelling, and hydrophilic and hydrophobic surface qualities. The swelling capacity of hydrogels is a crucial characteristic that is contingent upon the type of cross-linking present in the polymer networks. Solute diffusion to the hydrogel structure is made possible by the hydrogels' swelling capacity and porous nature. Hydrogels that are super-absorbent can

quickly absorb water and in amounts greater than 100% of their bulk (Lakshika Weerasundara et al., 2020).

1.1.4 CLASSIFICATION OF HYDROGEL

Since there are multiple hydrogel synthesis methodologies accessible for their manufacture, beginning with different types of raw materials, such as monomers and cross-linkers, there are different classification systems for hydrogels. In general, sources, polymeric composition, configuration, cross-linking type, physical appearance, network electrical charges, and polymerization techniques can all be used to classify something (Ahmed, 2015). Even when making hydrogels on an industrial basis, acrylic acid monomers are employed to make the majority of the hydrogels that are used for water treatment and other applications (Ahmed, 2015; Ahmed et al., 2013; Ali et al., 2003; Barakat & Sahiner, 2008; Kasgoz et al., 2003; Sun et al., 2016).

Table 1.2 Different hydrogels used for contamination removal in water and their functional groups (Lakshika Weerasundara et al., 2020)

Hydrogel	Available functional groups	Reference
Poly(acrylamide) hydrogel	-OH, N-H, -COOH, -CONH ₂	Sanyang et al. (2016)
Poly(acrylamide) hydrogel-rice husk biochar composite	-OH, -COO ⁻ , ether(C-O) groups, CH ₂ -O-Si	Sanyang et al. (2016)
Modified polyacrylamide hydrogels	-C=O, -NH ₂ , -CH ₂ , -NH	Kasgoz et al. (2003)
Magnetic cationic hydrogel	-C=O, -C-N, -[N ⁺ (CH ₃) ₃]	Tang et al. (2010)
Fe ₃ O ₄ nanoparticle functionalized polyvinyl alcohol/chitosan magnetic composite hydrogel	-OH, -CH ₃ , -C-O-C, -NH ₂	Yan et al. (2018)

Acid hydrolysis lignin-g-poly- (acrylic acid) hydrogel	-OH, -C-O, -C-O-C	Sun et al. (2016)
Poly (N- hydroxymethyl acrylamide) hydrogel	-C=O, hydroxymethyl group, amide group	Kasgoz et al. (2006)

1.2 OBJECTIVES

1. Synthesis of Hydrogel

A chitosan-based hydrogel was created by mixing chitosan with a sodium stearate or sodium laureth sulfate solution and heating the mixture to 80 degrees Celsius while stirring it mechanically. Tiny amounts of glacial acetic acid were added to the mixture. After mixing for twenty to twenty-five minutes, the solution and the solid layer were separated. The resulting solid was then allowed to cure at room temperature (Tetsuya Furuike et al., 2017).

2. Characterization of the Synthesised Hydrogel

The hydrogel that was created was thoroughly characterized using an array of analytical methods. FT-IR, or Fourier-transform infrared spectroscopy, was utilized to clarify the chemical makeup and functional groups contained in the hydrogel matrix. The molecular structure and interactions inside the hydrogel network were further elucidated through the application of high-resolution nuclear magnetic resonance spectroscopy, or H-NMR. Water absorption tests were also carried out to evaluate the hydrogel's ability to retain water and its swelling behavior in various environmental settings. Additionally, scanning electron microscopy (SEM) research was used to look into the hydrogel matrix's internal structure and morphological characteristics (Devashish Gokhale et al., 2022).

3. Conduction of the removal of micropollutant by Hydrogel

To evaluate the impacts on micropollutant uptake, hydrogels are made using a monomer solution in water with a set amount of photoinitiator (5% PI) and cross-linking agent (10%

PEGDA), but with a variable amount of surfactant. Experiments conducted on various uptake-regeneration cycles show that these microparticles can be employed for extended periods without experiencing a discernible decline in performance (Devashish Gokhale et al., 2022).

4. Monitoring of the initial and final concentration of micropollutants in wastewater

Using a UV-visible spectrometer, the target pollutants' starting and end concentration levels were measured to determine the hydrogel's effectiveness in encapsulating micropollutants from wastewater. The amount of pollutant elimination was ascertained by recording the absorbance spectra of the wastewater samples both before and after they were treated with the hydrogel. Additionally, precise quantification of the target micropollutants' levels in the wastewater samples was made possible by calibration curves created with known concentrations of the pollutants (Tetsuya Furuike et al., 2017).

1.3 SCOPE OF THE WORK

The goal of the research is to use micelle-laden hydrogels to create a unique approach to the enduring problem of micropollutants in wastewater. The scope combines knowledge in environmental engineering, materials science, and chemistry in an interdisciplinary manner. Initially, the study will concentrate on comprehending the characteristics and behavior of micelles to investigate their potential as effective carriers for the capture of micropollutants. Simultaneously, efforts will be focused on creating hydrogels that are best suited to contain these micelles, guaranteeing their compatibility, stability, and maximal capacity for the adsorption of pollutants. The objective of this phase is to determine the effectiveness of the hydrogel formulation in removing micropollutants and to fine-tune it through extensive experimentation and characterization.

The study will then move on to assessing the created micelle-laden hydrogel system's performance in both simulated and actual wastewater conditions. To evaluate variables including pollutant concentration, pH levels, and the presence of competing ions, extensive testing under various circumstances will be conducted during this phase. The objective is to verify the hydrogel's efficiency in limiting interference from other wastewater constituents and trapping micropollutants with selectivity by methodical investigation. In addition, possible regeneration procedures for the hydrogel will be investigated to improve its sustainability and reusability over several treatment cycles.

The project's last phase will concentrate on scalability and practical implications, considering factors like cost-effectiveness, production feasibility, and regulatory compliance. Assessing the technology's commercialization potential and incorporation into the current wastewater treatment infrastructure would require close collaboration with industry partners and stakeholders. Furthermore, a wider understanding and acceptance of the developed micelle-laden hydrogel technology will be facilitated by the transmission of knowledge through publications, workshops, and partnerships. In the end, the project seeks to promote a more resilient and resource-efficient water management system while developing sustainable wastewater treatment techniques and reducing the negative effects of micropollutants on the environment and human health.

CHAPTER 2

LITERATURE REVIEW

2.1 SYNTHESIS OF HYDROGEL

Chitosan-Based Hydrogels: From Preparation to Biomedical Applications: Pellá, M.C.G., M.K. Lima-Tenório, E.T. Tenório-Neto, M.R. Guilherme, E.C. Muniz, and A.F. Rubira

Over the past decade, there has been a significant increase in interest in developing hydrogels based on polysaccharides as intelligent biomaterials. Polysaccharides have been found to possess biocompatibility, biodegradability, non-toxicity, and affordability, making them promising candidates for biomaterial production. Hydrogels made of polysaccharides such as starch, carboxymethylcellulose, alginate, carrageenan, and chitosan are frequently used for biomaterials. Among these, chitosan has been extensively studied by biotechnology scientists due to its unique properties as a naturally occurring cationic and hydrophilic polymer. With its numerous benefits, including biocompatibility, biodegradability, and responsiveness, hydrogels are an excellent choice for the production of intelligent biomaterials, and polysaccharides are the key to their development. (Pellá et al. 2018)

Applications of Chitosan in Wastewater Treatment: Nechita, P

Chitosan is a remarkable material that boasts a range of attractive properties, including hydrophobicity, biocompatibility, biodegradability, and non-toxicity. Additionally, it contains highly reactive amino and hydroxyl groups in its backbone, which makes it an ideal adsorbent for removing pollutants from wastewater. But what sets chitosan apart from other polysaccharides is its unique chemical structure that allows for tailored alterations to create

polymers for specific uses. The reactive groups in chitosan can create composites with various substances, leading to superior adsorption capacities for wastewater contaminants that can withstand even the harshest acidic environments. Moreover, the cationic charge of chitosan effectively neutralizes and flocculates anionic-suspended colloidal particles, which results in lower levels of turbidity, chlorides, and chemical oxygen demand in wastewater. To sum up, chitosan's unique properties make it an ideal solution for wastewater pollution removal. Its versatility and effectiveness make it a valuable tool for dealing with water pollution, and its potential for tailored alterations to meet specific needs makes it a wise investment for any industry dealing with wastewater. (Nechita 2017)

Micelle-Laden Hydrogel Microparticles for the Removal of Hydrophobic Micropollutants from Water: Gokhale, D., I. Chen, and P.S. Doyle

Initial studies have shown that absorbent polymers can remove micropollutants from water. Micelles with hydrophilic shells and hydrophobic cores can extract hydrophobic compounds from water, which can be eliminated with simpler and more efficient methods like ultrafiltration. Hydrogel microparticles with chemically tethered micelles have been synthesized and demonstrated to be effective in water filtration. These bulk absorbents are easier to extract from water, regenerate, and have a wider range of chemical diversity.

The study focuses on synthesizing the micelle-laden hydrogel particles by creating an aqueous solution of monomers with a photo-initiator, a cross-linking agent, and one of the four acrylate surfactants. The surfactants, including B25MA, S80TA, T80TA, and F127DA, produce different sizes and architectures of particles. The surfactants' amphiphilic nature allows them to form micelles in the monomer solution, which are then integrated into the cross-linked hydrogel. Moreover, having density, the hydrogel particles seem easy to separate through simple gravity settling. Once the hydrogel particles have reached saturation in a water solution

containing 2-naphthol, the water is removed, and 90% ethanol is added to renew the hydrogel particles. To eliminate any remaining ethanol that can obstruct the quantification of 2-naphthol, the particles are combined with 90% ethanol and left in the dark for 24 hours. Another 24-hour wash follows this in clean water. After adding water containing naphthol, the equilibrium uptake is determined as previously mentioned. These uptake-regeneration cycles are extended to evaluate the effect of recurrent regeneration on hydrogel performance. (Gokhale et al. 2022)

2.2 CHARACTERISATION OF SYNTHESISED HYDROGEL

Preparation and Characterization of Hydrogels with Potential for Use as Biomaterials: Thürmer, M.B., C.E. Diehl, F.J.B. Brum, and L.A. dos Santos.

The hydrogels in this study were analyzed using FT-IR spectroscopy. The researchers obtained infrared spectra of the hydrogels using a Spectrum 1000 (PerkinElmer) spectrometer in the range of 400-4000 cm^{-1} with KBr discs. The IR spectrum of pure liquid NVP showed two very strong bands that are sensitive to the extent of the reaction. The first band, at 1629 cm^{-1} , is due to C=C bond stretching vibrations. The second, attributed to carbonyl stretching (C=O), is located at 1706 cm^{-1} . Greever et al. also identified characteristic IR absorptions for NVP at 1623 cm^{-1} (C=C) and 1700 cm^{-1} (C=O). The non-appearance of these bands indicates that the NVP monomer has polymerized, and a broad C=O bond appears at 1650 cm^{-1} in polyvinylpyrrolidone (PVP). The characteristic IR absorption at ~1630 cm^{-1} is assigned to the C=C bond stretching vibration of MBAM. The infrared region of 3800-3000 cm^{-1} is attributed to the OH stretch mode of water.

Consistent with the literature discussed above, the FTIR spectra of the crosslinked NVP films showed the disappearance of the characteristic NVP and MBAA absorptions, and the appearance of a broad C=O absorption, characteristic of PVP, at 1650 cm^{-1} . This suggests that

the conditions for polymerization were sufficient to essentially completely react the double bonds in the NVP and MBAA. The appearance of absorption in the region of 1550cm^{-1} (amide II band) suggests that the MBAM was successfully crosslinked with NVP during polymerization.

According to Devine and Higginbotham, the carbonyl group of PVP exhibits a stretching vibration peak between 1650 and 1680 cm^{-1} , and the carbonyl group of the carboxylic acid group on the PAA chain exhibits a peak at approximately 1750 cm^{-1} . When the carbonyl group forms an intermolecular bond (such as a hydrogen bond), there is a negative shift exhibited in the FTIR Spectrum, which can be observed in the M4 sample. It is also evident that a small shoulder appeared in this sample at about 1730 cm^{-1} corresponding to the stretching vibration of the carbonyl group of the carboxylic acid group on the PAA chain, which further illustrates that some intermolecular hydrogen bond did occur and the complexation formed between the PVP bead and PAA. Therefore, this suggests that the M4 sample may have formed a semi-interpenetrating network. (Thürmer et al. 2014)

2.3 REMOVAL OF MICROPOLLUTANTS FROM WASTEWATER USING HYDROGELS

Micelle-Laden Hydrogel Microparticles for the Removal of Hydrophobic Micropollutants from Water: Gokhale, D., I. Chen, and P.S. Doyle.

Hydrogels are created by mixing a monomer solution in water with a fixed amount of cross-linking agent (10% PEGDA) and photoinitiator (5% PI). However, the amount of surfactant used varies to assess its effect on micropollutant uptake. The partition coefficient, which is the ratio of the concentration of 2-naphthol inside and outside the particles, is found to be greater

than 1 for all systems. This indicates that hydrophobic micropollutants always preferentially partition into hydrogel microparticles. This partitioning is due to two effects: the physical separation of pollutant molecules into hydrophobic micelle cores and the formation of hydrogen bonds between the pollutant molecules and ethylene oxide units in the hydrogel matrix.

The partition coefficient increases when water is in the supernatant. This is because the increased number of micelles in the hydrogel increases its carrying capacity. The introduction of micelles disrupts hydrogen bonding in the hydrogel. However, the micelle-mediated effects dominate when water is the solvent. The partition coefficient of F127DA is higher than S80TA and B25MA, corresponding to the larger size of the hydrophobic zone in those molecules.

Micelle-laden hydrogels are used to clean contaminated water. Pollutants can be recovered from microparticles by washing them with a smaller volume of 90% ethanol. The materials studied in this work have higher mass transfer coefficients than commercial activated carbon. This makes the production of microparticles easy to scale up. The presence of micelles allows spent absorbent to be regenerated. The extent to which regeneration is possible increases with the amount of incorporated surfactant. Micelle-laden hydrogel microparticles are therefore a promising material for the elimination of hard-to-remove hydrophobic micropollutants from water.

CHAPTER 3

MATERIALS AND METHODOLOGY

Micelle-laden hydrogel is formed by the association of a surfactant and a cross-linking polymer. The fabrication of micelle-laden hydrogels is a multi-step process. It begins with the creation of micelles, tiny spheres formed by self-assembling molecules. These spheres then get incorporated into a three-dimensional network constructed from precursor molecules, creating the final micelle-laden hydrogel.

3.1 MATERIALS REQUIRED

- Chitosan
- Sodium Stearate
- Glacial Acetic Acid
- Sodium hydroxide
- Millipore water
- Micropollutant water sample



Figure 3.1 Sodium Stearate (Soap)



Figure 3.2 Chitosan and Glacial Acetic Acid



Figure 3.3 Micropollutant water sample

3.2 METHODOLOGY

3.2.1 SYNTHESIS OF MICELLE-LADEN HYDROGEL

The first step involves dissolving 1 gram of chitosan in 30 millilitres of water. To aid this process, 5 millilitres of acetic acid are added, triggering a deacetylation reaction that enhances chitosan's solubility. This mixture is heated to 85°C to facilitate dissolution. Next, we focus on micelle formation. Two separate solutions are prepared, one containing sodium stearate (soap) and another containing sodium laureth sulfate (SLES). To create the micelle-laden hydrogel, solution A (soap) or solution B (SLES) is mixed with the chitosan solution. This results in a dramatic transformation, forming a dense, solid, jelly-like structure. This jelly is separated from the remaining liquid, and voila, we have our micelle-laden hydrogel! Interestingly, repeating this process with both solution A and solution B yields two distinct hydrogel samples, each containing a different type of micelle. In evaluating the hydrogels formed from sodium stearate and SLES solutions, we opted for the sodium stearate hydrogel due to its superior structural characteristics and stability.



Figure 3.4 Mixing of the materials



Figure 3.5 Stirring the materials in oil bath



Figure 3.6 Experimental Setup



Figure 3.7 Hydrogel from Sodium Stearate



Figure 3.8 Hydrogel from SLES

3.2.2 REMOVAL OF METHYLENE BLUE DYE USING SYNTHESISED HYDROGEL

Methylene blue, a dye renowned for its striking blue colour, finds uses beyond vibrant textiles and even delves into the realm of medicine. However, this same dye can pose a significant threat when it infiltrates water sources. While the initial consequence might be a displeasing blue tinge to the water, the far greater concern lies in methylene blue's toxicity at elevated concentrations. This unwelcome presence disrupts the delicate equilibrium of aquatic ecosystems, hindering the vital processes of the organisms that call it home. The impact doesn't stop there, as the potential for harm extends to human health if contaminated water is ingested. This highlights the importance of proper wastewater treatment and environmental monitoring to safeguard our water resources from the detrimental effects of methylene blue contamination.\

A methylene blue dye solution was prepared, its vibrant blue hue a stark contrast to the desired clarity of clean water. The synthesized hydrogel, designed with a specific purpose in mind, was then carefully incorporated into the sample. After thorough mixing to ensure even distribution, the mixture was left undisturbed for 24 hours. This incubation period allowed for maximum contact between the dye molecules and the hydrogel's intricate network. The following day, the results were dramatic. The initially bright blue solution had undergone a tremendous colour change, hinting at a potential interaction between the dye and the hydrogel. This observation warranted further investigation to determine the extent of the hydrogel's ability to remove or alter the dye, offering valuable insights into its effectiveness for water treatment applications.

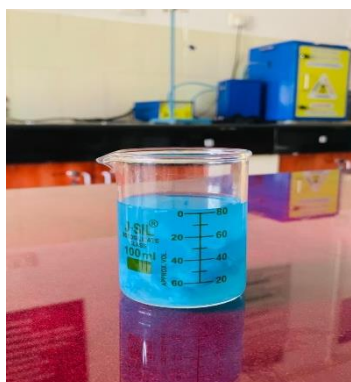


Figure 3.9 Hydrogel introduced to the water- dye solution

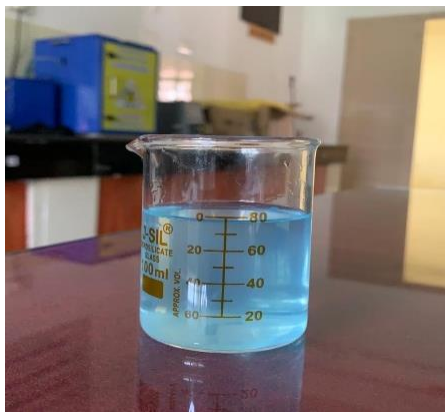


Figure 3.10 After 24 hours ,
Hydrogel- water- dye solution



Figure 3.11 Hydrogel containing dye

3.2.3 ENCAPSULATION OF MICROPOLLUTANT USING EMULSION-COACERVATION REACTION

Emulsion coacervation is a potent method of material separation and concentration. It is preparing an emulsion, usually oil in water, in which the target ingredient is encapsulated in oil droplets by the interaction of polymers. The enclosed material is squeezed and concentrated by driving the polymers closer together by the manipulation of variables such as pH and salt content. Through this procedure, the solution is divided into two phases: a diluted serum phase and a denser coacervate phase that is rich in the encapsulated substance. This adjustable method provides a flexible instrument for encapsulation, the production of microparticles, and focused separation in different domains.

The popular painkiller ibuprofen is regrettably also a cunning environmental monster. It belongs to the class of substances known as micropollutants, which are traces of pollutants that find their way into our water supplies. Ibuprofen is persistent, despite these tiny amounts being inconsequential. It can build up in bodies of water and is not easily broken down. This is concerning since even minute quantities might harm aquatic life and possibly human health.

The prepared hydrogel matrix is dispersed into the water sample containing micropollutants (Ibuprofen). Here, constant stirring is crucial to ensure even distribution. Next, we slowly introduce a sodium hydroxide (NaOH) solution to the mixture, gradually increasing the pH. This triggers an interesting phenomenon: the formation of a dense coacervate phase. Imagine tiny droplets of micropollutants being trapped within this dense, jelly-like substance. Our goal is to isolate this coacervate phase, which acts like a microscopic net capturing the unwanted pollutants. We can achieve this separation through filtration or centrifugation, effectively removing the coacervate from the bulk water. Finally, the collected coacervate undergoes a purification step to eliminate any unbound materials, ensuring we have a clean and concentrated sample of the captured micropollutants. This process offers exciting possibilities for water treatment, allowing for the targeted removal of contaminants.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 CHARACTERISATION OF HYDROGEL BY FT-IR

The generated hydrogel's FTIR examination provides important information about its molecular makeup and structure. FTIR spectroscopy verifies the successful creation of the intended polymeric network by identifying distinctive absorption peaks that correspond to particular functional groups within the hydrogel, such as OH, C=O, and NH bonds. Furthermore, shifts or variations in peak intensities show interactions between the hydrogel's constituent parts, offering important details about the material's overall stability and crosslinking effectiveness.

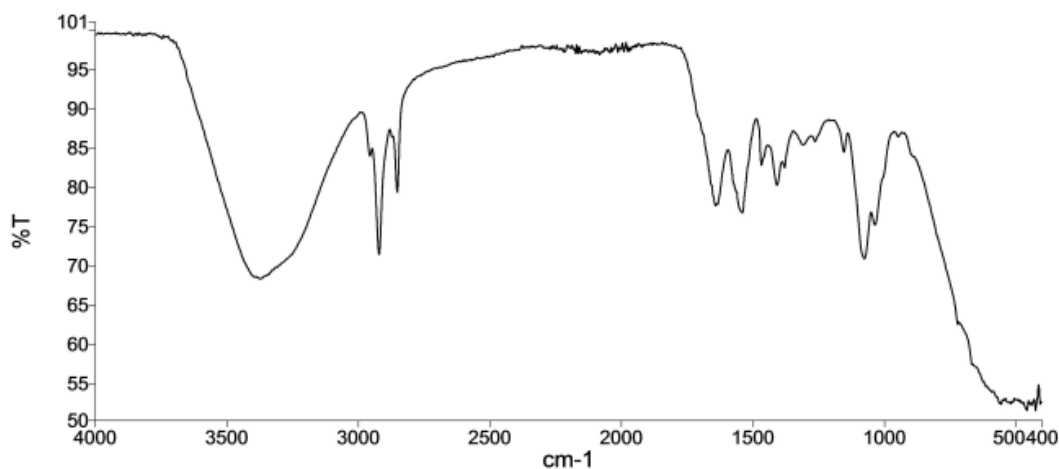


Figure 4.1 FT-IR of synthesised micelle hydrogel

4.1.1 FT-IR ANALYSIS FOR MICELLE HYDROGEL

The micelle hydrogel's examination reveals important molecular properties that are necessary to comprehend its structure and composition. Significant O-H stretching is indicated by the large peak seen at 3300 cm^{-1} , which points to the presence of hydroxyl groups—presumably

from water molecules or hydroxyl functional groups within the hydrogel matrix. In addition, strong peaks in the range of $2900\text{--}3000\text{ cm}^{-1}$ indicate aliphatic C–H stretching, which may be the result of hydrocarbon chains in the micelles or other organic components. Together, these spectral characteristics highlight the hydrogel's organic nature and offer some preliminary information on its chemical composition.

Of particular note is the prominent peak at approximately $1700\text{--}1750\text{ cm}^{-1}$, which indicates C=O stretching and perhaps carbonyl groups from amides, carboxylic acids, or esters in the hydrogel network. Furthermore, C–O stretching is correlated with peaks in the $1000\text{--}1300\text{ cm}^{-1}$ range, indicating the existence of ether linkages or other oxygen-containing compounds.

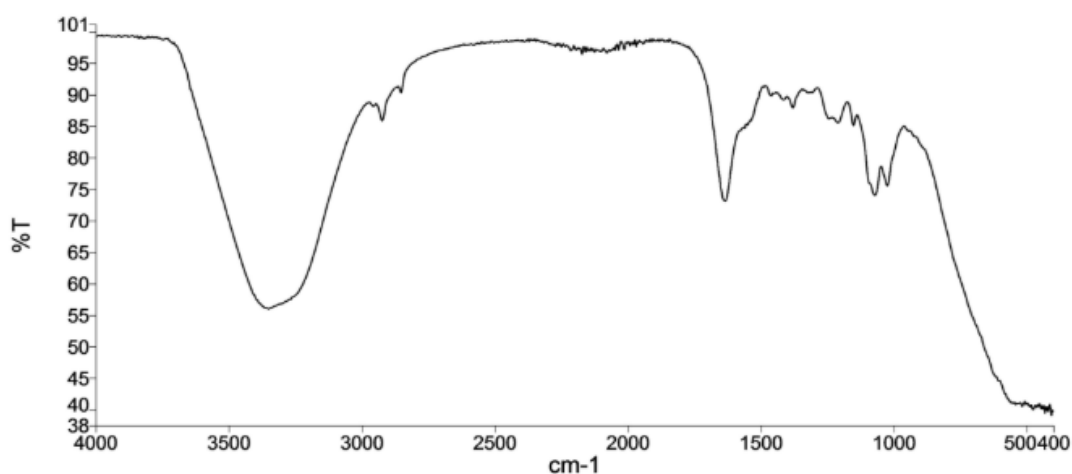


Figure 4.2 FT-IR of synthesised SLES hydrogel

4.1.2 FT-IR ANALYSIS FOR SLES HYDROGEL

Critical understanding of the molecular makeup and structure of the SLES hydrogel is provided by the examination of the hydrogel. Considerable O–H stretching vibrations are indicated by the large peak seen at 3300 cm^{-1} , which suggests the existence of hydroxyl groups, most likely from water molecules or hydroxyl functional groups within the hydrogel matrix. Sharp peaks in the $2900\text{--}3000\text{ cm}^{-1}$ region also indicate aliphatic C–H stretching vibrations, which could

originate from the hydrocarbon chains in the SLES molecules or from other organic components in the hydrogel.

The prominent peak at around 1700–1750 cm^{-1} represents C=O stretching vibrations, which may represent carbonyl groups derived from amides, carboxylic acids, or esters in the SLES hydrogel. Furthermore, C-O stretching vibrations are correlated with peaks in the 1000–1300 cm^{-1} range, indicating the possibility of ether linkages or other oxygen-containing moieties.

4.2 UV ANALYSIS OF IBUPROFEN

A quantitative analysis of ibuprofen micropollutant is performed using UV spectroscopy. This method involves creating a calibration curve using known concentrations of ibuprofen standards and then measuring the concentration of an unknown sample.

Table 4.1 Data of UV analysis of Ibuprofen

Name	Type	Concentration (mg/L)	AU (222.00 nm)	Dilution Factor	Original Conc. (mg/L)
Standard 1	Standard	0.00	0.0014		
Standard 2	Standard	5.00	0.083		
Standard 3	Standard	10.00	0.1394		
Standard 4	Standard	15.00	0.2031		

Standard 5	Standard	20.00	0.2342		
Sample 1	Sample	9.38	0.125	1	9.38
Sample 2	Sample	9.81	0.13	1	9.81

Primarily, four solutions with known ibuprofen concentrations are prepared. These standard solutions typically range from 5 ppm (parts per million) to 20 ppm in our case. Each standard solution is then scanned using a UV spectrophotometer. The instrument measures the amount of light absorbed by the ibuprofen molecules at a specific wavelength, typically around 222 nm for ibuprofen. By plotting the absorbance values against their corresponding concentrations, a calibration curve is established. This curve represents the relationship between the absorbance of ibuprofen and its concentration.

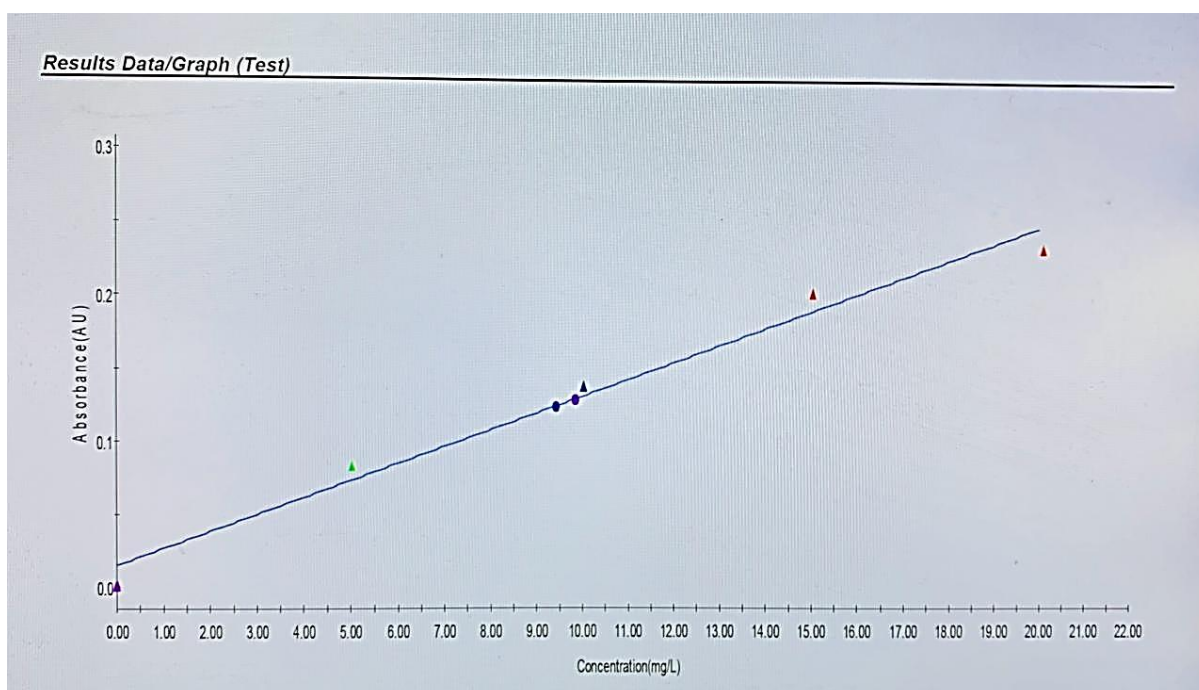


Figure 4.3 Absorbance vs Concentration curve of Ibuprofen

A micropollutant water sample containing an unknown concentration of ibuprofen (around 15 ppm in this scenario) is used. This sample undergoes an encapsulation process using a hydrogel. The specific details of this encapsulation process would depend on the chosen method, but it likely involves trapping the ibuprofen molecules within the hydrogel matrix.

After encapsulation, the final solution is measured again using UV spectroscopy. The absorbance value is then compared to the calibration curve to determine the unknown concentration of ibuprofen in the original water sample. By referencing the corresponding concentration on the curve based on the measured absorbance, the final ibuprofen concentration after encapsulation can be quantified. This encapsulation efficiency is calculated as follows:

Initial Concentration of water sample = 15 ppm

Final Concentration of water sample = 9.38 ppm

Efficiency = [(Initial conc- Final conc) / Initial conc] x 100

$$= \frac{15-9.38}{15} \times 100$$

$$= 37.47\%$$

This approach allows us to not only quantify the initial ibuprofen concentration in the water sample but also assess the impact of the encapsulation process on the overall concentration.



Figure 4.4 Different concentration samples

CHAPTER 5

CONCLUSIONS

A potential remedy for the problems associated with environmental pollution is the creation of a hydrogel that is loaded with micelles and intended to remove microparticles from wastewater. Using chitosan and sodium stearate or sodium laureth sulfate solution, we have created a hydrogel that is a versatile and efficient adsorbent material. FTIR spectroscopy characterization validates the hydrogel structure's effective development and its use in waste water treatment procedures. Furthermore, using UV-visible spectrometer measurement, the hydrogel's effectiveness in eliminating micropollutants like bisphenol as well as dye (methylene blue) from wastewater has been shown, highlighting its adaptability and efficiency to a variety of contaminants.

Future developments in this field might entail examining the micelle-laden hydrogel's scalability, long-term stability, and performance in various environmental settings. Furthermore, determining the financial viability and ecological consequences of incorporating this hydrogel-based technology into current wastewater treatment systems would be crucial for its larger-scale practical application. All things considered, the creation and use of such cutting-edge materials represent a critical advancement toward environmentally sound water management techniques and the protection of the environment for coming generations.

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