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Synthesis and Characterization of a Molecularly Imprinted Polymer Membrane for Selective Adsorption of 2-Phenylphenol from Aqueous Systems

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ABSTRACT

Modern agriculture increasingly relies on synthetic and natural fungicides, which can accumulate in environmental compartments, particularly soil and water. These residues and their degradation products are now recognized globally as significant contaminants, posing risks to aquatic organisms, terrestrial life, and human health depending on their concentration and chemical nature. Among emerging technologies for pollutant removal, molecularly imprinted polymers (MIPs) have gained attention due to their high selectivity, cost-effectiveness, ease of preparation, and large adsorption capacity. MIPs function as artificial receptors with strong affinity for specific target molecules. In this study, 2-phenylphenol (2-PP), a widely used fungicide, was selected as the target compound. MIPs were synthesized via

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precipitation polymerization in acetonitrile using 2-PP as the template, styrene as the functional monomer, divinyl benzene as the cross-linker, and azobisisobutyronitrile as the initiator. The prepared MIP particles were incorporated into polystyrene to fabricate a molecularly imprinted membrane (MIM). Characterization of the membrane was carried out using Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), X-ray diffraction (XRD), and Brunauer-Emmett-Teller (BET) analysis. The adsorption performance of the MIM was evaluated by studying kinetics, pH effects, and selectivity. Maximum adsorption was observed at pH 5 for MIM and pH 7 for non-imprinted membranes (NIP). Optimal contact time was 150 minutes, and the most effective adsorbent dose was 100 mg. The developed MIM demonstrated efficient removal of 2-PP from aqueous samples, indicating its potential application in water purification.

Keywords: Molecularly Imprinted Membrane (MIM), 2-Phenylphenol, Precipitation Polymerization, Adsorption Kinetics, Environmental Remediation

1. Introduction

2-phenylphenol is widely used as a fungicide to combat fungal diseases affecting crops. Its action involves breaking down fungal cell membranes so it helps in the inhibition of growth and limits its expansion. Their characteristic against fungicide extends to various commercial applications like preservation of wood, food preservation and processing of textile is due to its effectiveness against a broad range of fungi. But Improper disposal or excessive use of materials containing 2-phenylphenol (2-PP) can contaminate water and soil and damage ecosystems and harm beneficial insects and aquatic organisms (Hosoya et al., 2019).

Additionally, 2-phenylphenol persist in the environment for longer period of time and accumulate in soil and water systems causing long-term biological effects. Effective management techniques and regulatory oversight are critical to minimize the negative environmental effects of 2-phenylphenol. The Environmental Protection Agency has established strict regulations to stop the concentration of this compound in drinking water. Analytical methods have also been developed to monitor the presence in environmental samples due to due to known toxicity and carcinogenic potential (Pal & Kioka, 2025).

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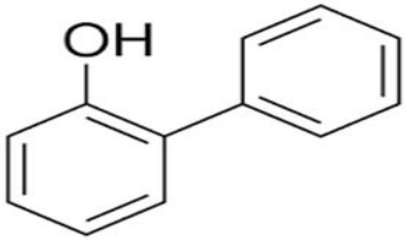

Figure 1.1: Structure of 2-Phenyl Phenol

The wider use and efficiency, some concerns about potential hazards are also related with 2-phenylphenol. Presence of 2-PP in products such as citrus fruits and its uses in pesticides rise questions about its impact on the environment as well as on human health. Controlling the quality of present water sources is a major challenge, especially in relation to the presence of fungicides like phenyl phenol, which can enter water systems through industrial effluents and imperfectly at in wastewater treatment plants. Minimizing the overall impact requires careful consideration and sustained efforts. This needs interagency cooperation, synthesis for sustainable practices that enhances environmental management and public health (Shen et al., 2023).

1.2 Molecularly Imprinted Polymers (MIPs

MIPs denotes a major advancement in polymer science, characterized by their bio-mimetic properties and three-dimensional shape formation around a target molecule, or template. They exhibit exceptional selectivity for spatial structure and binding sites, but holds great promise for applications that require selective binding like affinity sensors, separation, and catalysis (BelBruno, 2018).

Traditional methods for the production of MIPs rely heavily on organic chemicals, raising environmental concerns. However, the advent of green techniques such as ionic liquids, microwaves, ultrasonic technology, and supercritical carbon dioxide has provided clean methods for the preparation of MIP, through increased homogeneity and morphology, and organic solvents a reduced use. Environmentally friendly methods offer promise for nano-to-micron applications of MIP. Molecular imprinting involves the creation of particular regions in polymeric materials that support template molecules through complex interactions between active monomers and target materials, facilitated by cross-linkers and porogen solvents once the template is removed, the resulting cavities in MIPs selectively target molecules. It is

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triggered by various interactions such as hydrophobic, or dipolar interactions (Shen et al., 2023).

There are many methods for the synthesis of MIPs, including non-covalent, covalent and semi-covalent methods, each of which differs in the nature of the template and the active monomer polymer-binding sites in the final product interact. These processes include bulk polymerization, emulsion, multistage swelling, gelation, precipitation processes such suspension, resulting in particles ranging from irregular to perfectly spherical, and nano- to micron-sized (Viveiros et al., 2018).

Depending on the intended application, MIPs can be obtained in various forms such as membranes, fibers, core and shell particles. Compared to other purification methods, MIP synthesis is less time-consuming and cost-effective, making it attractive for applications requiring selective drying of the surface MIP epitope MIPs synthesized have overcome challenges of addressing template extraction efficiency, detection and speed, extending their usefulness in sampling, drug sensors, controlled release, and enantiomer separation (Pichon et al., 2019).

2-Phenylphenol is broadly used fungicide. Because 2-phenylphenol can be released into the environment, it has been related to a variety of adverse effects on humans. Although its significance in a range of industries, 2-phenylphenol (2-PP) extraction and purification encounter hurdles in terms of selection, efficiency, and environmental impact. Molecularly imprinted polymeric membranes (MIMs) are an alternate method for removing 2-PP from complicated composites in the application. This constraint has to be studied and remedied. The main objective is to develop MIM-based extraction methods that exhibit enhanced selectivity, improved extraction efficiency, and reduced environmental impact compared to conventional methods. Therefore, it is important to prepare specific extraction agents for 2-phenylphenol. This study will be based on the identification of structures and properties of MIMs specially optimized for 2-PP detection, followed by evaluation of their performance in terms of binding capacity, selectivity and recyclability will be, as it can be used in industries such as agriculture, pharmaceuticals and food processing.

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2. Review of Literature

The growing pollution of water resources with organic substances, especially fungicides, 2-phenylphenol (2-PP), in particular, has become a significant environmental issue. These are persistent, toxic and capable of accumulating in the aquatic environment with risks to human health and biodiversity. Traditional water treatment process is usually nonselective and ineffective in eliminating such micro pollutants, which has given rise to development of advanced materials like molecularly imprinted polymers (MIPs).

Molecular imprinting is another method that is employed to form polymeric materials that contain certain recognition sites that the target molecules are recognized by. The idea was first formulated in the early 20th century though it received great attention after the work of Wulff and Mosbach in the 1970s who demonstrated how to synthesize polymers with predetermined selectivity (Trotta et al., 2012).

MIPs are prepared by polymerizing functional monomers and cross-linkers in the presence of a template molecule which is subsequently removed, leaving behind cavities that are complementary in shape and functionality to the target molecule. These materials are a replica of natural receptors like antibodies, but have greater chemical stability, reduced cost and can be reused (Vo et al., 2024).

MIPs have been integrated in the last few years with membrane technology to create an MIMs which combines selective recognition with continuous separation processes. Membranes are selective barriers regulating the movement of molecules between stages, and the presence of imprinting sites increases their selectivity to certain analytes (Saylan et al., 2019).

The application of MIMs to water treatment, chemical sensing and separation technologies has shown promising applications that selectively adsorb and separate target molecules in complex mixtures (Ratnaningsih et al., 2022).

Various fabrication techniques have been designed in MIMs, such as in situ polymerization, phase inversion, and creation of composite membranes. These strategies are expected to maximize membrane permeability, selectivity and mechanical stability. As per earlier research, the effectiveness of MIMs is determined

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by template-monomer interactions, cross-linking density, and morphology of the membrane (Lu et al., 2019).

Complex techniques of characterization like FTIR, SEM, XRD and BET analysis are widely applied to the analysis of structural and surface properties of these materials. Recent developments indicate the use of MIMs in remediation of the environment especially in the removal of organic pollutants in water. It has been demonstrated that MIMs have high adsorption capacity, rapid kinetics, and high selectivity in contrast to non-imprinted polymers (NIPs) (Torres-Cartas et al., 2020).

Their selectivity to target molecules despite the presence of structurally similar molecules makes them highly suitable in selective adsorption processes. Although these benefits are present, there are still certain drawbacks, such as the inability to completely remove the template, the lack of access to the binding sites, and difficulties in large-scale production. Current research is aimed at optimizing the synthesis methods, at optimizing the work of the membranes, and at studying new applications in the environment and industry. Altogether, molecularly imprinted polymer membranes are one of the promising and effective methods of selective removal of contaminants like 2-phenylphenol in aqueous media, and should be considered in the development of sustainable technologies of water purification

3.Materials and Methods

3.1. Chemicals and Reagents

All chemicals used in this study included 2-phenylphenol (2-PP), styrene, polystyrene, tetrahydrofuran (THF), distilled water, acetic acid, acetonitrile, AIBN, divinyl benzene (DVB), and acetone. These chemicals were of analytical grade and obtained from Sigma-Aldrich.

3.2. Instrumentation

The following instruments were used for synthesis and characterization: centrifuge, shaker, bath sonicator, UV–Visible spectrophotometer, FTIR spectrometer, scanning electron microscope (SEM), BET analyzer, and water bath.

3.3.Preparation of Standard and Working Solutions

A stock solution (100 ppm) of 2-PP was prepared by dissolving 0.2 g in 1000 mL distilled water. Working solutions (5–30 ppm) were prepared by dilution. The

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maximum absorbance (λmax) of 2-PP was observed at 272 nm using a UV spectrophotometer.

3.4. Synthesis of MIM and NIM

Molecularly imprinted membranes (MIMs) were synthesized using non-covalent precipitation polymerization. The template (2-PP) was mixed with acetonitrile and sonicated for 30 minutes. Styrene (monomer), DVB (cross-linker), and AIBN (initiator) were added, and the mixture was purged with nitrogen to prevent oxidation. Polymerization was carried out in a water bath at 80°C for 8 hours, followed by 85°C for 4 hours. The product was collected by centrifugation.

Non-imprinted membranes (NIMs) were prepared using the same method without adding the template.

3.5. Membrane Fabrication

MIM particles were embedded into polystyrene membranes using the phase inversion method. Polystyrene was dissolved in THF, and MIM particles were added and stirred at 50°C for 4 hours. The solution was cast onto a glass plate and dried overnight. NIM membranes were prepared similarly.

3.6.Characterization

The prepared membranes were characterized using FTIR, SEM, BET, UV–Vis spectroscopy, and XRD to determine their structural, morphological, and chemical properties.

3.7.Batch Binding Studies

Batch adsorption experiments were conducted using 2-PP solutions. Membranes (MIM and NIM) were added to the solution and stirred. Samples were taken at regular intervals, and absorbance was measured using a UV spectrophotometer. Binding efficiency was calculated as:

$$Q\ (\%) = Ci - \frac{Cf}{Ci} \times 100$$

3.8.Effect of Polymer Dosage

Different amounts of MIM (100–500 mg) were added to 2-PP solution to study the effect of adsorbent dosage, keeping other parameters constant.

3.9.Effect of pH

The pH of the solution (3–13) was adjusted using HCl and NaOH. The effect of pH

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on binding efficiency was studied under constant conditions of time, concentration, and temperature.

3.10.Effect of Contact Time

The effect of contact time (30-270 minutes) on adsorption was studied using fixed conditions of pH, concentration, and membrane dosage. Residual 2-PP concentration was measured after each interval.

4.Results and Discussions

4.1.Preparatory Conditions for Polymerization of 2-PP

Several important factors must be considered when designing Molecularly Imprinted Membranes (MIMs). First of all, a suitable alloy was selected that will provide a polymer matrix with greater porosity. After that a strong bond must be created in between the template and the monomer to achieve a best product. The synthesis of a highly porous structure and mechanical strength are largely dependent on the cross-linking monomer. For effective printing and removal, the kind and quantity of active monomer utilized must be carefully chosen. At the end, stability and ideal quality of the resultant MIM are guaranteed by managing the polymerization conditions. By careful consideration of these variables, researchers created MIMs that improved extraction performance by having a stronger binding capability for the specified template monomer (Mayer et al., 2019). Styrene proved as the monomer and divinylbenzene (DVB) used as the cross-linker in this study. A molecularly imprinted membrane (MIM) having greater adsorption for the 2-phenyl phenol (2PP), was produced by the mixing cross-linker and the monomer. For the study of effects of various factors, like adsorption conditions, and applications in river water samples, a cross linker of molar ratio 16 was used due to its good affinity and selectivity (Bhavani et al., 2018). To enhance the development of monomeric complexes, some more amount of styrene was used than 2-PP. Styrene molecules and 2-PP created noncovalent contacts. This resulted in the generation of both electrostatic and non-electrostatic forces. The quantity of functional groups per repeating unit, or amount of active binding regions in the 2-PP molecules, was used to determine the degree of binding. Styrene's benzene ring allowed for easier π-π stacking, but 2-PP molecules hydroxyl group made it harder. provided sites, which

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contributed to the selective recognition of MIMs The planned mechanism for 2-PP-MIM synthesis involves the incorporation of one 2-PP molecule and four styrene molecules by π-π stacking, followed by confinement of the polymer matrix as cross-linking monomers using DVB as cross linker.

4.2.Batch Binding Analysis of 2PP-MIM

Batch binding of both MIM and NIM was performed to select the most efficient MIM. MIM binding capacity was determined by using batch mode binding procedures. MIM showed the highest efficiency compared to NIM as shown in graph 4.1, because MIM has a higher molar ratio of functional monomer which would provide particular binding regions and so will increase the binding capacity. The MIM reached the greater binding ability at 150 minutes indicating that the adsorption procedure was not slow. More expansion upon exposure did not result in any productive change in the binding power and remained about the similar due to saturation. For NIM, the capacity is lower due to lack of connected binding cavities.

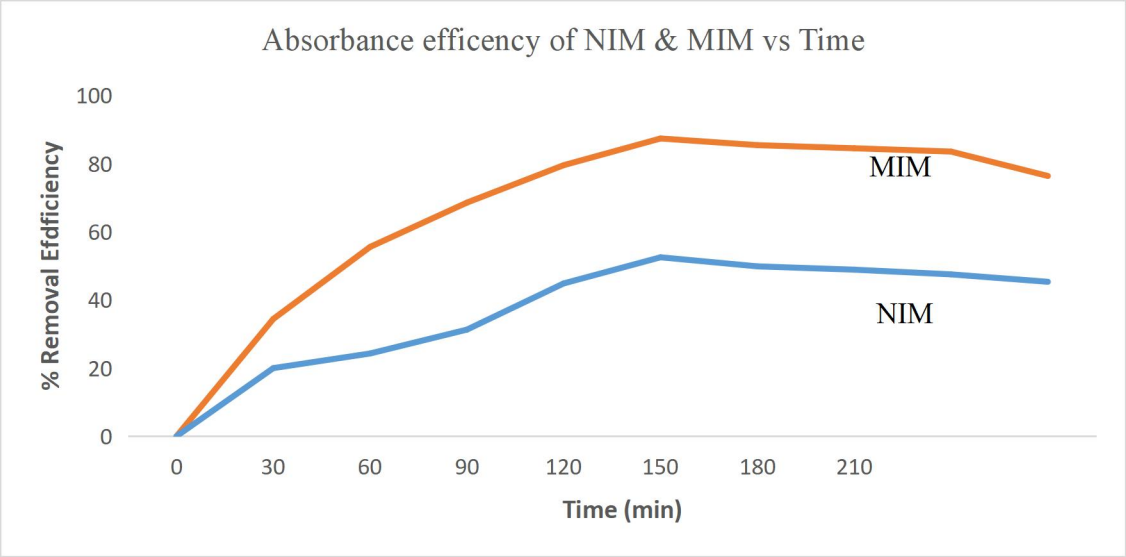

Figure.4.1: Absorbance efficiency of NIM & MIM vs time

4.3. Effect of Initial Concentration on Binding Efficiency of MIM

For the adsorption study, the experimental work was conducted at various concentrations of 2-Phenyl Phenol (10, 20, 30, 40 ppm). Figure 2 indicated that binding efficiency of MIM at start increased with increased polymer concentration of template (10 ppm to 30ppm) from 37% to 78% and then decreased from 78 % to 60% in the concentration of template (30 ppm to 40 ppm). Maximum efficiency

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(approximately 78 %) was observed at 30 ppm Senthilkumar et al, (2005) reported in a study reported that MIM binding efficiency first increased sequentially from 87% to 99% in the concentration of the template (15 ppm - 25 ppm) and then decreased from 90% to 86 % in the concentration of the template (30 ppm - 40 ppm). The maximum binding efficiency of 99% was obtained at 25 ppm and this could be due to the optimal number of binding cavities on the MIM surface. More increase in concentration of template may oversaturate the surrounding MIM and was fewer binding sites are also present were recorded. MIM shows higher efficiency and better specificity at higher concentrations of template molecules while MIM binding efficiencies are characterized by high-affinity binding sites (Foo & Hameed, 2012).

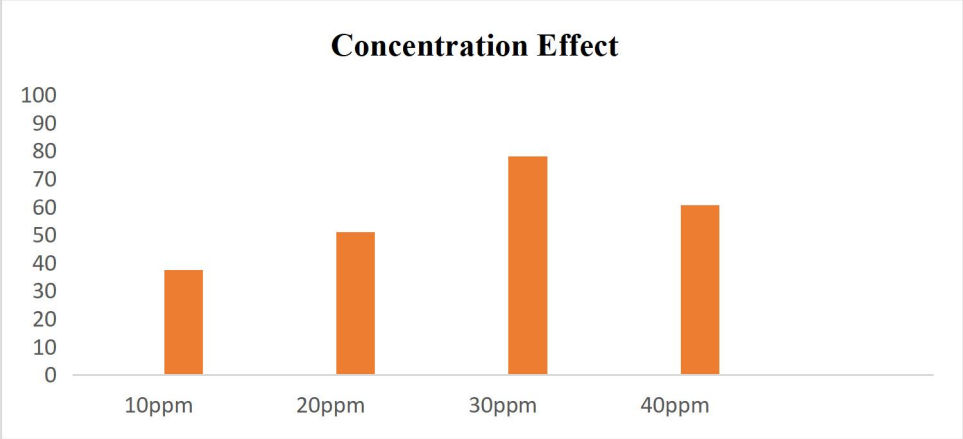

Figure. 4.2 Absorbance efficiency of MIM vs concentration

4.4.MIM Dosage Effect on Binding Efficiencies of 2-PP MIMs

Different polymer concentrations were used to determine the binding efficiency. Figure 4.3 clearly shows that the binding efficiency has decreased with increasing polymer concentration. This could be due to the accumulation of polymer fragments (Pathania et al., 2017). This is due to the reason that they have reduced the number of active binding regions available because of self-assembly of the polymer fragments. Binding capacity decreased with increasing polymer concentration. Mohan et al., (2001) and Taha et al., (2009) presented the results of their study in which the impact of adsorbent amount on binding efficiency was determined. Figure below 4.3 syndicates the highest binding efficiency of 2PP-MIPs occurs at 0.2 g while the binding efficiency decreases slightly after that because of aggregation of MIM.

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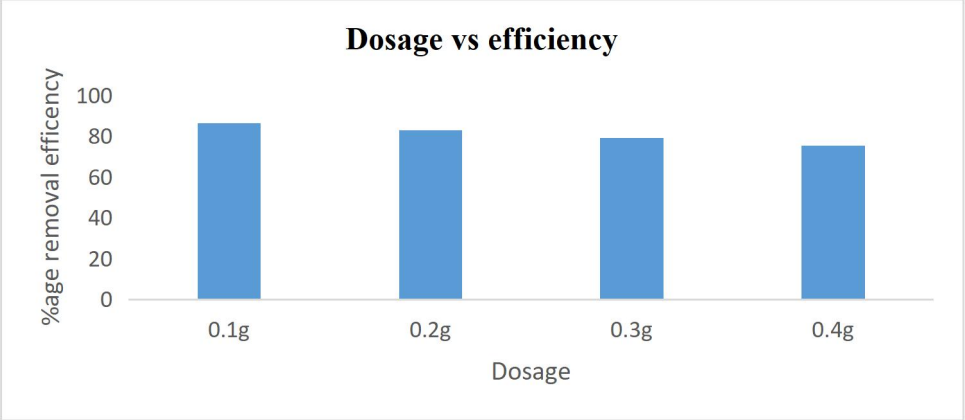

Figure.4.3: Absorbance efficiency of MIM vs dosage

4.5. Effects of Time contact on the Binding Efficiency of 2PP MIMs

The contact time studies were recorded in the range of 30-270 min keeping the rest of the parameters constant. The results obtained are presented in figure 4.4. The results obtained indicate that 2PP adsorption was extremely fast in the initial 150 min and approximately 99 percent of the 2PP was eluted due to the emergence of more active binding sites on the surface of MIP. The adsorption process became nearly constant after 150 min and the graph shows no further changes of the 2PP adsorption at MIP surface. The physical capability of the adsorption method was demonstrated in the form of at start rapid 2PP binding by the MIP (Farooq et al., 2010). The graph flattened after 150 min, which indicates that there was no significant change in the binding efficiency of 2PP by MIP. Equilibrium time is defined as the time at which 2PP-MIP can achieve maximum binding efficiency of 150 min. Various researches on adsorbents indicate that each adsorbent possesses equilibrium time at which they reach peak efficiency and beyond that they reach equilibrium (Mohammed et al., 2014). The binding effectiveness, according to the curve in Figure 4.4, increased rapidly initially, and peaked at the specified initial concentration within 150 minutes. The binding of 2PP on MIPs involves two stages: there is a fast binding and a slow uptake of the 2PP by MIPs. Also, during adsorption of 2PP, initially the molecules arrive at the boundary layer and subsequently, such molecules need to adsorb into the surface of absorbent and finally, they need to enter into the porous structure of MIP (Iscen et al., 2007). The first was that initially binding was very fast when the binding sites of MIP far exceeded the

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number of template molecules in the bulk of the liquid. Consequently, this will necessitate a relatively extended contact time of this phenomena. The same tendency has been reported by Senthilkumar (2018) and other Researchers.

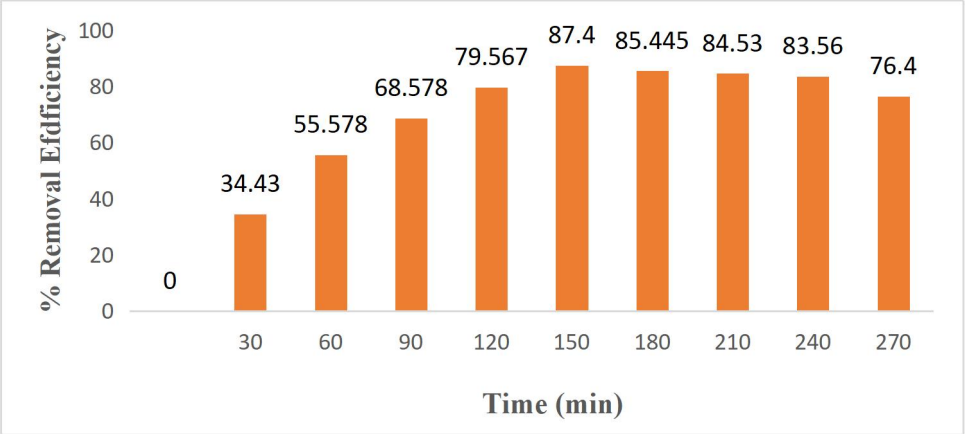

Figure. 4.4: Removal efficiency of MIM vs time

4.6. FTIR analysis of NIPM

The FTIR spectrum of the polymer membrane which was not imprinted indicates that there are several functional groups. The wider peak of approximately 3369 cm -1 is probably as a result of the hydroxyl group stretching vibrations. Even though it has approximately a peak at 2922 cm -1 at C-H cm -1 stretching mode of vibrations, which point to the presence of aliphatic hydrocarbon chains. The maximum at 1725cm -1 might be attributed to C-O stretching vibration but might be due to traces of acetone molecules present. The non-imprinted polymer membrane FTIR study is provided in although the peaks of about 1459 cm-1 and 1489 cm-1 may be regarded as C=C stretching and it indicates the presence of aromatic rings. Figure 4.5. The fact that the lower wavelength peaks indicated that there were both amine and carbonyl groups. These functional groups determine the properties of polymers.

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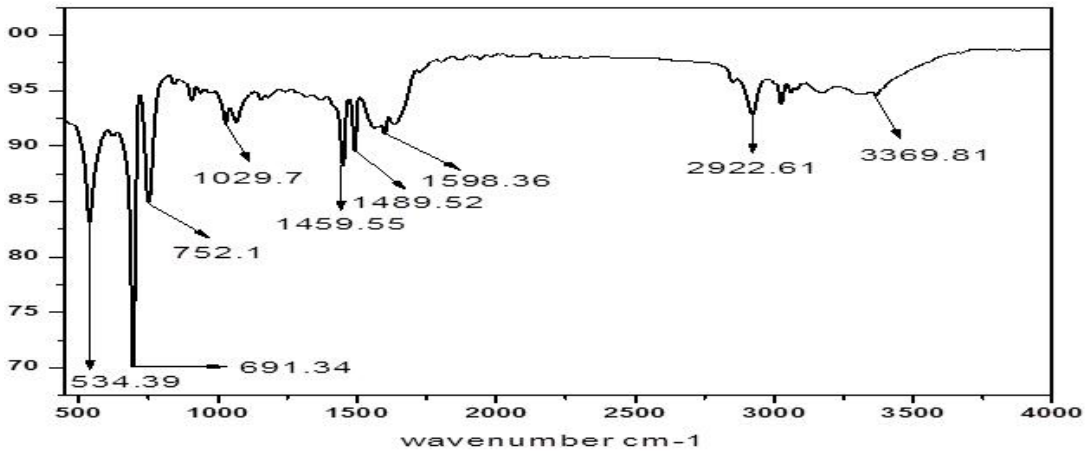

Figure. 4. 5: FTIR analysis of non-imprinted polymer membrane

4.7.2-PP Molecularly Imprinted Polymer Membrane FTIR Analysis

The FTIR spectra confirmed the effective synthesis of 2-PP by showing the typical peaks of 2-PP. The peak at 3400 cm-1 indicated the presence of O-H stretching vibrations. The other peaks at 2982 cm -1 corresponds to the C-H stretching vibrations which promised the presence of aliphatic hydrocarbon chains. The presence of amine and carbonyl groups correlates with other peaks at lower wavenumbers which was observed through C-O stretching vibrations (approximately 1725.3 cm -1) and C-N stretching vibrations (approximately 1398 cm -1). These functional groups and the specific binding sites emerged in the imprinting process and this also contributed to the selectivity of the polymer and the capability to recognize 2- Phenyl phenol.

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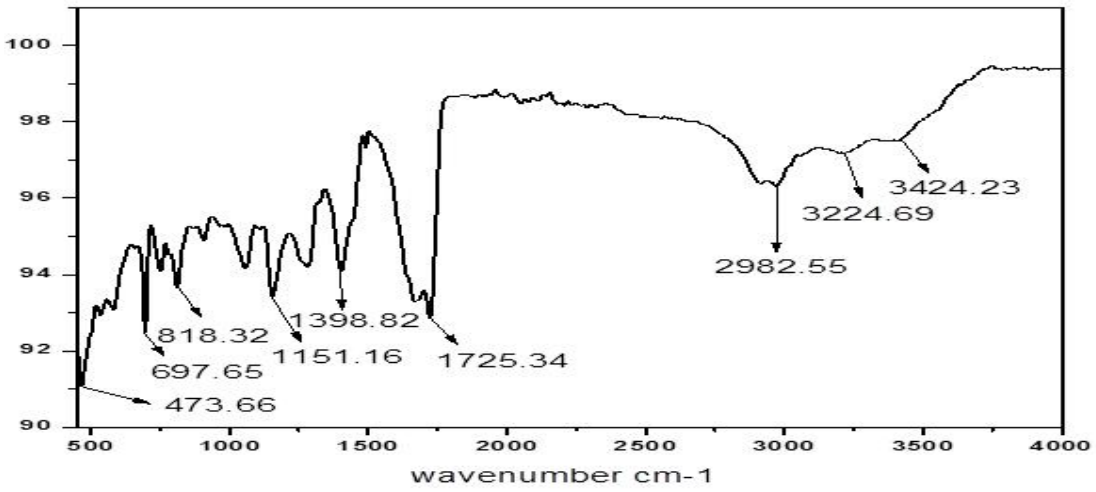

Figure.4.6: FTIR analysis of 2-PP Molecularly Imprinted Polymer Membrane

4.8.XRD Spectra of 2-Phenyl Phenol Molecularly Imprinted Membrane

In the XRD spectrum of the 2-phenylphenol molecularly imprinted membrane, a 22°, 42o, 62o and 82o peak were observed. They are primarily exhibited by the polymeric crystal structure and appearance of imprinted holes. This peak was observed at 22, and the crystalline structure of the polymer matrix of the membrane is connected to this peak. It verified the presence of parts in the polymer. The maximum at 42 o could be due to the development of imprinted substance. The exact location of surface can also be influenced by the morphology of the imprinted cavity. The peaks at 62 o and 82 o can also be related to other crystalline components of the membrane or crystalline structure of the polymer. These peaks will mean that the molecularly imprinted membranes are relatively ordered in shape and that voids are created which will be capable of binding 2-phenyl phenol molecules efficiently, during imprinting.

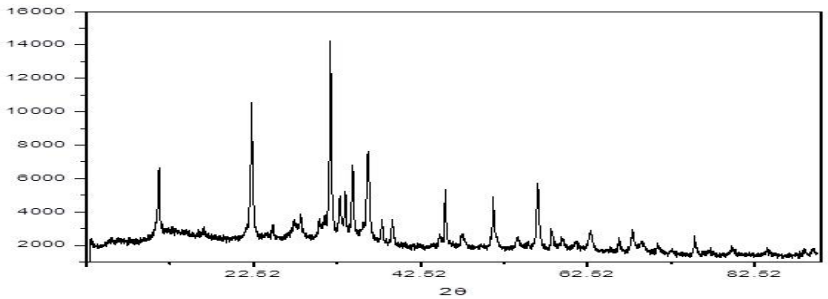

Fig.4.7: XRD Spectra of 2-Phenyl Phenol Molecularly Imprinted Membrane

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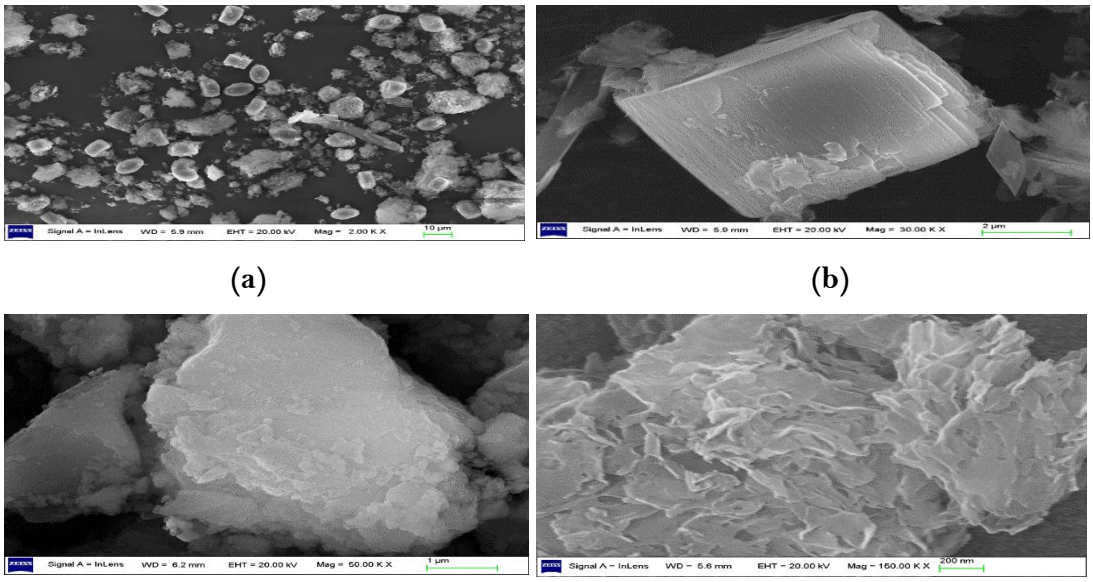
4.9.SEM Analysis of NIM vs MIM

The SEM images show that there are morphological differences between the molecularly imprinted membrane (MIM) and the non-imprinted membrane (NIM). The MIM is more particulate with possible imprinted cavities and surface roughness indicating that the template molecule had an effect on the formation of the polymer. By comparison NIM exhibits a more aggregated, layered structure with less discrete particles. These changes are probably affecting the surface area, porosity and finally binding selectivity and target molecule binding capacity of the membranes. The MIM particulate morphology might enable particular interactions in the imprinted sites whereas the structure of the NIM is non-specific binding.



(a) (b)

(c) (d)

Figure. 8 SEM analysis of NIM vs MIM (a) SEM of NIM (b) Magnified SEM image of NIM (c) SEM of MIM (d) Magnified SEM image of MIM

4.10. BET of MIM and NIM

The diameter of the pore, volume of pore and unique surface area (m2/g) of NIM and 2PP-MIM are listed in Table 8. The results indicated that MIM was superior to NIM in all measures, as well as specific surface area and the volume of the pore. As shown in the results of table 4.8, the presence of a template in the process of MIM formation created a cavity and blocked the capabilities of the pores to decrease during the polymerization process. Consequently, MIM contained bigger pores

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compared to NIM. The results of the BET also indicated the creation of an appropriate dimensional morphology of the binding of the template by MIM. BET analysis will also indicate the presence or absence of a template during the synthesis process, potentially giving a possible solution to selectively eliminate templates in different samples. MIMs were washed and analyzed by BET to ascertain the presence of porous cavities that the template molecule had left behind. ACN was selected as solvent during the polymerization process, contributes to making MIM particles porous, as the BET values in Table 4.8 show. According to Esfandyari-Manesh et al. (2021), ACN was a preferred pyrogenic solvent that imparts a higher level of porosity to the MIM. The data in Table 4.8 show that MIM has a higher surface area compared to NIM. This means that the 2-PP template molecule that is contained in MIM helps in forming and enhancing the porous nature of the polymer.

Table. 4.8: ***BET results of 2PP-MIM and NIM***

Properties	2-PP-MIM	NIM
Surface area (m^2/g)	133.22	19.735
Mean Radius of pore (Å)	8.767	4.4353
Total volume of the pore ($cm^3 g^{-1}$)	5.01	3.33

5. Conclusion

The most advanced technique is now being considered to be the creation of different recognition media through selective adsorption through molecular imprinting. The research and development of artificial receptors appears to be of interest. The emphasis of this work is the efficient production of polymer membranes with molecular imprints that are designed to target a particular target molecule, 2-phenylphenol. To selectively eliminate its templates in the test materials, the resulting molecularly imprinted polymer membrane did a fantastic job. MIM have been successfully used as sorbents to selectively extract 2-phenylphenol fungicides. MIM of 2-PP was prepared through the use of the precipitation polymerization method. MIM microspheres evolved by Precipitation polymerization and with uniform sizes and shapes. This method produced imprinted polymer membranes of 2-phenylphenol that formed the uniform size and form. A lot of research was done to investigate the impact of agitation rate, pH, initial concentration, MIM dose and contact time. It was established that the size, shape and degree of interaction of the

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binding site with the target molecule were key determinants of MIM selectivity. The MIM also exhibited greater binding efficiency and selectivity towards 2-phenylphenol. SEM, FTIR and BET have been used to characterize MIM. The selective regeneration of 2-PP was done using the developed MIM on test materials once it was optimized. The results of the selectivity studies show that the actual synthesized MIM possesses a more selective adsorption capability in comparison to the other structural homologue of 2-PP. My work will assist in the future research on the extraction procedure of 2-PP by the use of MIMs. Consequently, in this paper the positive extraction efficiency of MIP synthesized would suggest the successful development and optimization of these MIM.

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