GREEN MAGNETIC MOLECULARLY IMPRINTED POLYMER FOR SELECTIVE REMOVAL OF PARABENS FROM COSMETIC SAMPLES

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To my beloved mother and father, thank you very much for your moral support during my difficult time. The keys to every triumph and achievement I have attained are your love, encouragement, and day and night prayers.

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ABSTRACT

Parabens are chemicals that are frequently utilized as preservatives in cosmetic samples. In recent years, the safety concern over these compounds has grown due to their endocrine-disrupting activity. In this research, a green magnetic molecularly imprinted polymer (GMMIP) was synthesized and applied as an adsorbent to selectively recognize and remove parabens from cosmetic samples by using propylparaben as a template. The green strategies of GMMIP were introduced by the utilization of *Kesum* leaves extract as a reducing agent in the synthesis of green magnetic nanoparticles (MNP) as a magnetic core and by designing deep eutectic solvent (DES) as an environmentally friendly functional monomer in the preparation of the GMMIP. As a reference, a green magnetic non-imprinted polymer (GMNIP) was also synthesized without propylparaben. This study was focused on the characterization, adsorption, and application of GMMIP. The obtained GMMIP was characterized by Fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscopy (FESEM), x-ray powder diffraction (XRD), thermal gravimetric analysis (TGA), vibrating sample magnetometer (VSM), and Brunauer-Emmett-Teller (BET). The characterization results proved that the GMMIP exhibited an irregular spherical shape, mesoporous characteristics with a pore size of 17.74 nm, and magnetic characteristics. The selective adsorption experiments indicated that GMMIP showed significant selectivity for propylparaben than methylparaben and ethylparaben. The GMMIP exhibited the highest adsorption capacity (2.7608 mg/g) and a good imprinting factor (2.63) on propylparaben than GMNIP. The adsorption processes appeared to best fit the pseudo-second-order kinetic and Freundlich isotherm models at an optimum pH of 12. The thermodynamic results also indicated that the adsorption process was exothermic, spontaneous, and more favorable at 298 K. The optimized GMMIP was applied as an adsorbent in the selective extraction of parabens from real cosmetic samples. The results demonstrated good recoveries ranging from 43.3-113.3 %. It was found that the limits of detection (LOD) and quantification (LOQ) were between 0.03 and 0.05 mg/L and 0.11 and 0.16 mg/L, respectively. The developed GMMIP was discovered to be a convenient and effective adsorbent for the extraction of parabens from cosmetic products.

ABSTRAK

Paraben adalah bahan kimia yang sering digunakan sebagai pengawet dalam sampel kosmetik. Dalam beberapa tahun kebelakangan ini, kebimbangan keselamatan terhadap sebatian ini telah berkembang disebabkan oleh aktiviti yang mengganggu sistem endokrin. Dalam penyelidikan ini, polimer tercetak molekul magnetik hijau (GMMIP) telah disintesis dan digunakan sebagai penjerap untuk mengenali secara terpilih dan mengekstrak paraben daripada sampel kosmetik dengan menggunakan propilparaben sebagai templat. Strategi hijau GMMIP diperkenalkan melalui penggunaan ekstrak daun Kesum sebagai agen penurunan dalam sintesis nanopartikel magnetik hijau (MNP) sebagai teras magnet dan dengan mereka bentuk pelarut eutektik dalam (DES) sebagai monomer berfungsi yang mesra alam dalam penyediaan GMMIP. Sebagai rujukan, polimer tidak bercetak magnet hijau (GMNIP) juga disintesis tanpa propilparaben. Kajian ini tertumpu kepada pencirian, penjerapan, dan aplikasi GMMIP. GMMIP yang diperolehi dicirikan oleh spektroskopi inframerah transformasi Fourier (FTIR), mikroskop elektron pengimbasan pelepasan medan (FESEM), pembiasan sinar-x (XRD), analisis gravimetrik terma (TGA), magnetometer sampel bergetar (VSM), dan Brunauer-Emmett-Teller (BET). Hasil pencirian membuktikan bahawa GMMIP mempamerkan bentuk sfera yang tidak teratur, ciri mesopori dengan saiz liang 17.74 nm, dan ciri magnet. Eksperimen penjerapan terpilih menunjukkan bahawa GMMIP menunjukkan selektiviti yang ketara untuk propilparaben berbanding metilparaben dan etilparaben. GMMIP mempamerkan kapasiti penjerapan tertinggi (2.7608 mg/g) dan faktor cetakan yang baik (2.63) pada propilparaben berbanding GMNIP. Proses penjerapan berlaku secara optimum dengan model kinetik pseudo tertib kedua dan isoterma Freundlich pada pH 12. Keputusan termodinamik juga menunjukkan bahawa proses penjerapan adalah eksotermik, spontan, dan lebih baik pada suhu 298 K. GMMIP yang dioptimumkan digunakan sebagai penjerap dalam pengekstrakan terpilih paraben daripada sampel kosmetik sebenar. Keputusan kajian menunjukkan pemulihan yang baik antara 43.3-113.3 %. Keputusan kajian juga mendapati bahawa had pengesanan (LOD) dan kuantifikasi (LOQ) masing-masing adalah antara 0.03 dan 0.05 mg/L dan 0.11 dan 0.16 mg/L. Secara keseluruhannya, GMMIP yang dibangunkan didapati sesuai sebagai penjerap yang mudah dan berkesan untuk pengekstrakan paraben daripada produk kosmetik.

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LIST OF ABBREVIATIONS

ATRP	-	Atom transfer radical polymerization
BET	-	Brunauer-Emmett-Teller
BJH	-	Barrett-Joyner-Halenda
CE	-	Capillary electrophoresis
CEC	-	Capillary electrochromatography
CPE	-	Cloud point extraction
DAD	-	Diode array detector
DES	-	Deep eutectic solvent
DLLME	-	Dispersive liquid-liquid microextraction
EDC	-	Endocrine disruption compound
FESEM	-	Field emission scanning electron microscopy
FRP	-	Free radical polymerization
FTIR	-	Fourier transform infrared
GMMIP	-	Green magnetic molecularly imprinted polymer
GMNIP	-	Green magnetic non-imprinted polymer
HBA	-	Hydrogen bond acceptor
HBD	-	Hydrogen bond donor

HPCE	-	High performance capillary electrophoresis
HPLC	-	High-performance liquid chromatography
HPLC-UV	-	High-performance liquid chromatography with ultraviolet detector
LLE	-	Liquid-liquid extraction
LOD	-	Limit of detection
LOQ	-	Limit of quantification
MIP	-	Molecularly imprinted polymer
MIT	-	Molecular imprinting technology
MISPE	-	Molecularly imprinted solid-phase extraction
MNP	-	Magnetic nanoparticles
MMIP	-	Magnetic molecularly imprinted polymer
MMIP-SPE	-	Magnetic molecularly imprinted polymer solid-phase extraction
MSPD	-	Matrix solid phase dispersion
MSPE	-	Magnetic solid-phase extraction
NMP	-	Nitroxide-mediated polymerization
NP	-	Nanoparticle
RAFT	-	Reversible addition-fragment chain transfer polymerization
RSD	-	Residual standard deviation
RTIL	-	Room temperature ionic liquids
SPE	-	Solid phase extraction
UAE	-	Ultrasonic-assisted extraction

UV-VIS	-	Ultraviolet-visible spectrometer
VA-DLLE	-	Vortex-assisted dispersive liquid-liquid extraction
VSM	-	Vibrating sample magnetometer
XRD	-	X-ray diffractometer

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Parabens are known as esters of para-hydroxybenzoic acid that are often used as preservatives and antimicrobial reagents in cosmetic products such as deodorants, eye make-up, lotions, shampoos, and scrubs (Garner *et al*., 2014). The most common parabens that are extensively used in cosmetics include methylparaben, ethylparaben, propylparaben, and butylparaben (Kirchoff & Gannes, 2013). However, recent research has indicated that parabens are classified as an endocrine-disrupting chemical (EDC) since they can harm both human and animal health by disturbing the endocrine system (Lincho *et al*., 2021). Another study revealed that paraben exposure can harm the reproductive system (Alkafajy & Abdul-Jabbar, 2020). Additionally, it has been established that parabens may cause breast cancer (Hager *et al*., 2022). These days, sample preparation techniques are crucial in determining the presence of parabens in cosmetics, particularly those with a complex matrix. Numerous separation techniques, including solid-phase extraction (SPE), solid-phase microextraction (SPME), dispersive liquid-liquid microextraction (DLLME), and ultrasonic-assisted extraction (UAE), have been developed. However, several of these techniques have limitations, including being time-consuming, laborious, and using a lot of hazardous solvents (Piao *et al*., 2014). Therefore, preconcentration methods are crucial methods implemented for sample preparation prior to the separation techniques mentioned above.

Molecularly imprinted polymers (MIPs) have recently evolved into a promising material for the preconcentration, identification, and removal of several types of molecules. Molecularly imprinted polymer (MIP) is an artificial polymer formed by introducing the target molecule (template) and subsequently being removed by an appropriate solvent (Cui *et al*., 2020). As a result, the unique cavities obtained are matched to the target molecule in terms of functional group, size, and structure (Chen *et al*., 2016). MIPs can detect and bind the template molecule given by the presence of imprinted cavities, making it possible to separate and detect the template molecule (López et al., 2020). MIPs have numerous benefits over conventional bioligands such as antibodies, aptamers, and lectins, including ease of production, good stability, and a relatively low cost (Belbruno, 2019). Nevertheless, traditional MIPs have a number of drawbacks and continue to utilize a significant volume of organic solvents, which results in higher production costs (Viveiros *et al*., 2018).

The contribution of the green magnetic nanoparticles approach has emerged with many advantages for molecular imprinting technology (MIT), such as small in size, biocompatibility, low toxicity, and excellent magnetic properties, which can improve some of the fundamental characteristics of MIP (Tabaraki & Sadeghinejad, 2020). Recently, the green synthesis of magnetic nanoparticles by biological method has gained a lot of interest since it has numerous advantages over chemical and physical methods, including being easy to prepare, cost-effective, and producing less waste (Yew *et al*., 2020). Magnetite nanoparticles, Fe_3O_4, have emerged as a potential candidate for study among all magnetic nanoparticles because of their superparamagnetic characteristics, biocompatibility, and low toxicity (Macías-Martínez *et al*., 2016). There have been numerous reports of successful studies employing different plant extracts for MNP synthesis, such as *Zanthoxylum armatum* DC (Ramesh *et al*., 2018), *Dolichos lablab* L (Basavaiah *et al*., 2018), *Kappaphycus alvarezii* (Yew *et al*., 2016), *Calliandra haematocephala* (Sirdeshpande *et al*., 2018), and *Lagenaria siceraria* (Kanagasubbulakshmi & Kadirvelu, 2017). Fortunately, the use of *Persicaria odorata* extract, commonly known as *Kesum* leaves extract is not documented in any literature to synthesize MNP. Hence, the aqueous leaf extract of *Kesum* is used in this research to fabricate Fe_3O_4 nanoparticles for the first time in a simple and eco-friendly manner.

On the other side, there has been an increase in interest in using deep eutectic solvent (DES) as a green functional monomer in the synthesis of MIPs (Li & Row, 2017;

Liu *et al*., 2020). DES provides many advantages, such as having a wide range of polarity, low volatility and toxicity, water-miscible, and biodegradable. A specific DES can be synthesized by changing the hydrogen bond donor (HBD) or hydrogen bond acceptor (HBA), which boosts the specific recognition of MIP compared to conventional MIP. Numerous investigations have shown that the combination of DES with a functional monomer aids in improving the selectivity and binding of the target molecules (Jablonský *et al*., 2020).

Therefore, the main goal of this work is to synthesize green magnetic molecularly imprinted polymer (GMMIP) and apply it as an adsorbent for the removal of parabens from cosmetic samples. The green magnetic nanoparticles were first synthesized using *Kesum* leaves extract as a reducing agent via the co-precipitation method. The green magnetic nanoparticles were then used as the magnetic core in the synthesis of GMMIP. Besides that, the DES was also introduced as an eco-friendly functional monomer to accomplish the green aspect of the GMMIP synthesis. The characteristics, selectivity, and binding properties of GMMIP were all investigated, and the extracted parabens were subjected to UV-VIS analysis.

1.2 Problem statement

In order to maintain cosmetic efficacy and create long shelf life, the addition of chemical additives in cosmetics products are inevitable. Parabens are the most popular preservatives that have been used in cosmetic products for over 50 years and they are thought to be safe and harmless (Fonseca *et al*., 2016). The four parabens that are most frequently found in cosmetics are methylparaben, ethylparaben, propylparaben, and butylparaben. Nevertheless, several reports have been published regarding the toxicity of parabens to human health (Bilal & Iqbal, 2019) and environment (Haman *et al*., 2015). In addition, parabens have been found to have potentially endocrine-disrupting compounds (EDCs) in humans and animals that can interrupt the endocrine system (Jagne *et al*., 2016). On the other hand, parabens are among a long list of substances that are either regulated or prohibited from usage in cosmetics. It was determined that parabens may induce breast cancer as a result of extended dermal exposure to deodorants that contained parabens (González-Mariño *et al*., 2011).

Due to the concerns about this toxicity and the continuous use of parabens, several extraction techniques and analytical studies have been conducted to remove parabens from cosmetic samples in order to measure their content. Multiple methods, such as solid-phase extraction (SPE), ultrasonic-assisted extraction (UAE), matrix solid phase dispersion (MSPD), and dispersive liquid-liquid microextraction (DLLME), have been used to extract parabens from complicated matrix. However, a number of them demand a lot of hazardous chemical solvents, labor-intensive, and time-consuming (You *et al*., 2016). Recently, MIPs have been seen as sophisticated materials that are designed to recognize and extract organic or inorganic analytes in many complex matrices owing to their high affinity and selectivity properties for their target compounds (Madikizela *et al*., 2018). However, in most situations, the reagents employed to produce MIPs are considered to be harmful and have a number of unavoidable problems, such as inadequate template removal, uneven distribution of binding sites, slow rebinding rates, and poor sensitivity (Liu & Dykstra, 2022).

In order to materialize the idea of "green chemistry", a green strategy was implemented, and MIP surfaces were modified. This is crucial for environmental conservation because it reduces the requirement for chemicals and circumvents the issues with conventional MIP. Furthermore, it is anticipated that incorporating magnetic nanoparticles into the MIP synthesis process will improve the properties of MIP and create the versatile material known as magnetic molecularly imprinted polymers (MMIPs). The MMIPs have outperformed the conventional MIP in terms of effective binding capacity, magnetic susceptibility, and simplifying the pretreatment procedure (Ariani *et al*., 2022). Therefore, in order to achieve the "green chemistry" concept in this study, *Kesum* leaves extract was used to produce green magnetic nanoparticles, and deep eutectic solvent (DES) was applied as a green functional monomer in the MIP synthesis. Thus, this has led to the study of green magnetic molecularly imprinted polymer (GMMIP) as the best candidate for the removal of parabens from cosmetic samples.

1.3 Research objectives

The following objectives are set forward for this study:

i. To synthesize and characterize the green magnetic molecularly imprinted polymer (GMMIP) nanoparticles by introducing green magnetic nanoparticles (MNPs) as a magnetic core and deep eutectic solvents (DES) as an eco-friendly functional monomer.
ii. To evaluate the performance of green magnetic molecularly imprinted polymer (GMMIP) nanoparticles for selective recognition of parabens.
iii. To apply and assess the performance of green magnetic molecularly imprinted polymer (GMMIP) nanoparticles as an adsorbent to remove the parabens from real cosmetics samples.

1.4 Significance of study

The significance of this study is the emerging role of green chemistry in the synthesis of green magnetic molecularly imprinted polymer (GMMIP). In this work, a green reagent which is *Kesum* leaves extract, was introduced as reducing agent in the synthesis of the green magnetic nanoparticles. In addition, deep eutectic solvent (DES) was also introduced as a green functional monomer in the preparation of GMMIP. Hazardous substance use can be reduced if these principles are followed. To date, only a few researchers have used benign chemicals for molecularly imprinted polymer (MIP) synthesis. This is a great potential field as new work emerges where the MIP is synthesized through the introduction of eco-friendly reagents. The effects of GMMIP will greatly benefit from cost reductions, including low-cost synthesis, reducing the quantity of chemical substances and toxic waste, and improving the safety of operators (Madikizela *et al*., 2018).

Furthermore, this work has the potential to improve and increase the selectivity of MIP properties, allowing it to overcome extraction limitations and reduce mass

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