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EFFECT OF GREEN POLYMERISATION VIA MICROWAVE-ASSISTED SYNTHESIS ON MORPHOLOGY, SIZE, AND REBINDING PROPERTIES OF ASPIRINATE MOLECULARLY IMPRINTED POLYMER

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Abstract: Molecularly Imprinted Polymer (MIP) has been vastly investigated for various applications including drug delivery systems. Precipitation polymerisation has been widely used in the preparation of MIP to obtain small-size polymer beads that are vital in drug delivery applications. However, the process requires a longer time and produces larger polymer particles which often appears as a constraint for large-scale production and drug delivery applications. In this study, a series of MIPs were synthesised via microwave (MW) irradiation employing aspirin as template in the present of methacrylic acid (MAA) as functional monomer and divinylbenzene as crosslinker in acetonitrile at 80°C (MW80 in 60 minutes), 100°C (MW100 in 15 minutes), 110°C (MW110 in 8 minutes), and 120°C (MW120 in 4 minutes). MW110 produced homogeneous spherical particles (1.24 µm ± 0.17 µm), higher yield (0.62 g), and excellent rebinding capacity (7.27 mg/g) compared with other MIPs. Green synthesis of MIP via MW emerged as a powerful tool for well-controlled, rapid reactions and is significantly useful for drug delivery applications.

Keywords: Drug delivery, molecularly imprinted polymer, microwave irradiation optimisation, microsphere, green synthesis.

Introduction

Molecularly Imprinted Polymers (MIPs) have garnered significant attention due to their exceptional selectivity and unique properties (Díaz-Álvarez *et al*., 2023) for a variety of fields such as environmental monitoring (Quinto *et al*., 2019; Li *et al*., 2020), food analysis (He *et al*., 2019; Aghoutane *et al*., 2020), biomedical diagnostics (Ayankojo *et al*., 2020), and drug delivery (Sajini *et al*., 2019; Jantarat *et al*., 2020). The MIPs are easily prepared via pre-polymerisation of the template and functional monomers complex, followed by co-polymerisation with crosslinker and the removal of the template a cavity (Samarth *et al*., 2015; Xiao *et al*., 2020).

Typically, bulk polymerisation, precipitation polymerisation, pickering emulsion polymerisation, multi-step swelling polymerisation, and surface imprinting processes are used in the conventional heating process of synthesising MIP (Ertürk & Mattiasson, 2017; Fresco-Cala *et al*., 2020). Nevertheless, the conventional method offers a longer reaction time (6 to 24 hours) and uneven heat distribution prompts the exploration of alternative approaches (Kueseng *et al*., 2018). In recent years, MW-assisted synthesis has emerged as an alternative and efficient technique for polymerisation (Lamaoui *et al*., 2021). MW-assisted synthesis is a fast and well-controlled reaction used extensively in organic synthesis and recently explored in polymerisation reactions (Brahmbhatt *et al*., 2020). The polymerisation of MIPs *via* MW has been reported as a rapid synthesis with lower energy consumption compared with conventional heating (Chen *et al*., 2015; Liang *et al*., 2018; Lamaoui *et al*., 2021; Zhang *et al*., 2021). The green synthesis of MIPs via MW irradiation has significantly influenced the overall performance, structural characteristics, morphology, shape, and binding capabilities of MIPs (Brahmbhatt *et al*., 2020). The excellent heat distribution in MW reaction system has offered rapid and uniform heating, thus, reducing the polymerisation time, improving energy consumption, and enhancing the MIP performances (Kueseng *et al*., 2018; Moret *et al*., 2019; Farooq *et al*., 2020).

In this study, MIPs were synthesised using aspirin (template), methacrylic acid, (MAA; functional monomer), and divinylbenzene (DVB; crosslinker) in acetonitrile as a porogenic solvent. The effect of green polymerisation via MW-assisted synthesis on the yield, morphology, size, and rebinding properties of the MIPs has also been evaluated. The use of MW irradiation for green polymerisation has great potential to advance the development of effective, sustainable, and tailored drug delivery systems with enhanced therapeutic outcomes.

Materials and Methods

Chemicals

Methacrylic acid, (99%, MAA), divinylbenzene (80%, DVB), 2,2’-azobisisobutyronitrile (AIBN) were obtained from Sigma Aldrich. Acros Organic provided acetylsalicylic acid 99%. Acetonitrile, glacial acetic acid, and methanol were obtained from Merck, Fisher Scientific and HmbG, respectively. All chemicals were used as it was delivered.

Instruments

The MW-assisted synthesis was carried out with Monowave 400 (Anton Paar GmbH). Particle morphologies were observed by a Field Emission Scanning Electron Microscope (FESEM) from (JSM-IT500HR, Electron Optics Laboratory Japan). ImageJ version 1.53t was used to measure the particle size. UV-1280 spectrophotometer (Shimadzu Corporation) was used to measure the UV-vis in a quartz cell (1.0 cm × 1.0 cm). The infrared spectra (400 cm^{-1} to 900 cm^{-1}) in potassium bromide (KBr) were recorded using Thermo Scientific Nicolet Model iS10 with Smart Omni-Transmission attachment.

Microwave Assisted Polymerisation

The MIPs were synthesised by precipitation polymerisation via MW-assisted synthesis. A mixture of 1 mmol of aspirin (0.18 g) and 4 mmol of MAA (0.34 mL) in acetonitrile (15 mL) was stirred at room temperature for 1 hour. Approximately 16 mmol of DVB (2.28 mL) and AIBN (3 mL) were poured into the solution mixture and nitrogen-degassed (15 minutes) prior to heat using Anton Paar Monowave 400 (Table 1). The produced MIP was subjected to filtration, washed with acetonitrile, and oven dried at 50°C for 18 hours. The polymer was exhaustively washed with methanol-acetic acid (9:1, v/v, 7 x 25 mL), followed by acetonitrile (4 x 25 mL) for template removal and monitored using UV-Vis.

Table 1: MW irradiation ratio for the preparation of MIP

Polymer	Aspirin (mmol)	MAA (mmol)	DVB (mmol)	Temperature	Time
MIP-C*	1	4	16	80°C	6 hours
MW80	1	4	16	80°C	60 minutes
MW100	1	4	16	100°C	15 minutes
MW110	1	4	16	110°C	8 minutes
MW120	1	4	16	120°C	4 minutes

*Conventional polymerisation of MIP (Lawai *et al*., 2024).

Rebinding Study

The dried MIP (80 mg) was dispersed in acetonitrile (20 mL) containing aspirin (100 mg/L). The mixture was shaken at 150 rpm at room temperature for 2 hours. UV/vis spectrophotometer was used to analyse the filtrate. The study was carried out in triplicate. Seven standard solutions of aspirin (10 mg/L to 110 mg/L) were plotted to obtain a calibration curve. Equation 1 was applied to calculate the MIP rebinding capacity (Q).

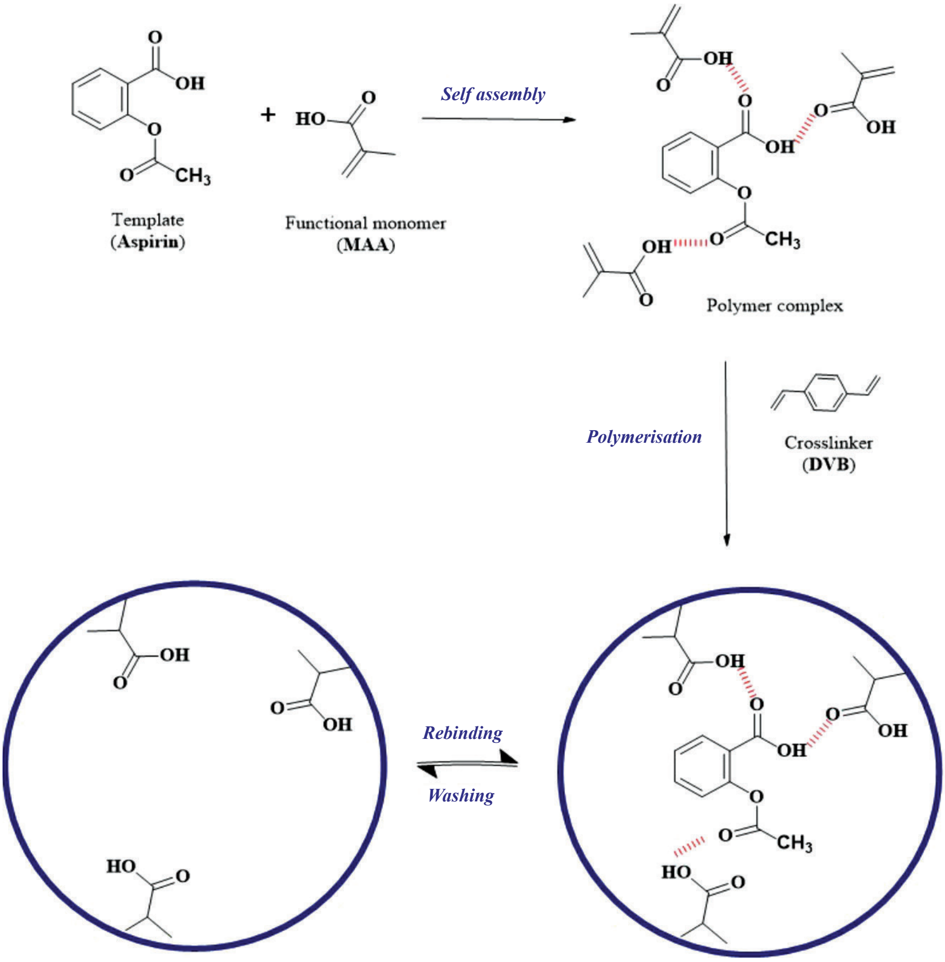
$$Q = \frac{V(Ci - Cf)}{m} \tag{1}$$

where Q (mg/g) is the number of aspirin bound, C_i and C_f (mg/L) are the initial and final aspirin concentration, V (L) is the volume of aspirin solution, and m (g) is the weight of MIP.

Results and Discussion

MW-assisted Synthesis of MIP

Scheme 1 illustrates the synthetic approach for the preparation of MIP. The pre-polymerisation complex was formed through non-covalent interactions between aspirin (template) and MAA (functional monomer). The copolymerisation occurred in the presence of DVB and AIBN. The formulation ratio of the template: functional monomer: crosslinker (1:4:16) was conducted



Scheme 1: Diagram illustrating the synthesis of MIP for aspirin

following earlier studies (Lawai *et al*., 2024a). The polymerisation of MIP was performed via MW irradiation with a series of temperatures at 80°C, 100°C, 110°C, and 120°C. The distinctive property of MAA as a functional monomer allows it to function as both a hydrogen donor and an acceptor of hydrogen bonds and exhibits good compatibility for ionic interactions (Lawai *et al*., 2024b). The MAA forms interaction with aspirin via hydrogen bonding from the carboxyl (C=O) and hydroxyl group (-OH). The DVB as a crosslinker had minimal interaction with the template, resulting in a decrease in non-specific binding site phenomena (Muhammad *et al*., 2012). The hydrogen bond interaction between the functional monomers and template molecules is often responsible for the molecular recognition of MIP (Chen *et al*., 2016; Włoch & Datta, 2019).

The Effect of MW Temperature on the Polymer Yield

The yield of MIP obtained via MW-assisted synthesis is shown in Figure 1. The yield of polymers obtained using MW is comparable with that of MIP produced via traditional thermal heating (MIP-C) in a shorter reaction time. The MW80 produced a higher yield (0.87 g), followed by MW110 (0.62 g), MW120 (0.58 g), and MW100 (0.53 g). Nevertheless, the low polymerisation temperature of MW80 at 1 hour produced a higher yield compared with MIP-C (6 hours). In a shorter polymerisation time, MW110 (8 minutes) showed a higher yield, compared with MW100 (15 minutes) and MW120 (4 minutes). The increase of the reaction rate *via* internal energy transfer by MW led to an increase in particle formation, enhancing both the yield and polymerisation time (Turner *et al*., 2010; Leadbeater, 2014; Shahar *et al*., 2016; Viveiros *et al*., 2018; Farooq *et al*., 2022).

Functional Group Characterisation of MIP

As shown in Figure 2, the prepared MIP at different temperatures were characterised using FTIR spectroscopy. All MIPs showed similar characteristic bands at different polymerisation temperatures. The polymer backbone consists of MAA and DVB. Broad -OH absorption bands were observed at 3,440 cm^{-1} to 3,455 cm^{-1}, which are attributed to the stretching vibration of O-H in the functional monomer MAA. The appearance of peak corresponded to C-H at 2,924 cm^{-1} to 2,928 cm^{-1} and the disappearance of peak attributed to C=C in MAA and DVB at 1,620 cm^{-1} to 1,680 cm^{-1}, indicates the occurrence of crosslinked polymerisation, of the allyl group presence in MAA and DVB [Figures 2 (a-d)] (Mustafai *et al*., 2018). This phenomenon occurs during free radical polymerisation, where the alkenes in MAA and DVB are formed in the presence of AIBN initiator and propagate the chain growth by the addition of monomer and crosslinker and formed a new single bond (Wloch & Datta, 2019). The peak corresponding to *v*C=O was observed at 1,700 cm^{-1} to 1,705 cm^{-1}.

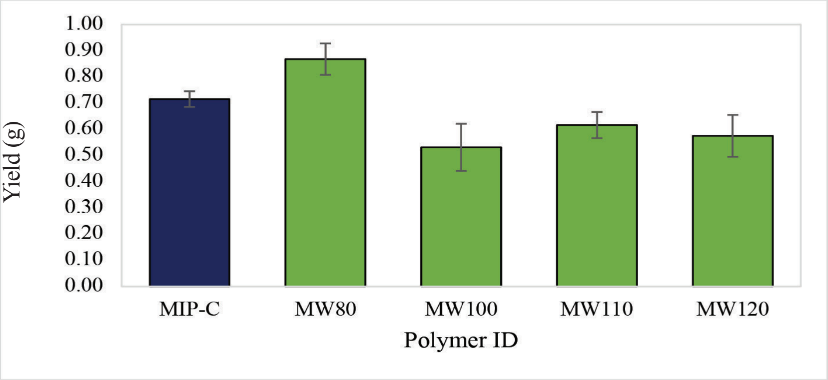


Figure 1: Yield of polymer produced by MW-assisted synthesis and conventional thermal heating (MIP-C) (Lawai *et al*., 2024)

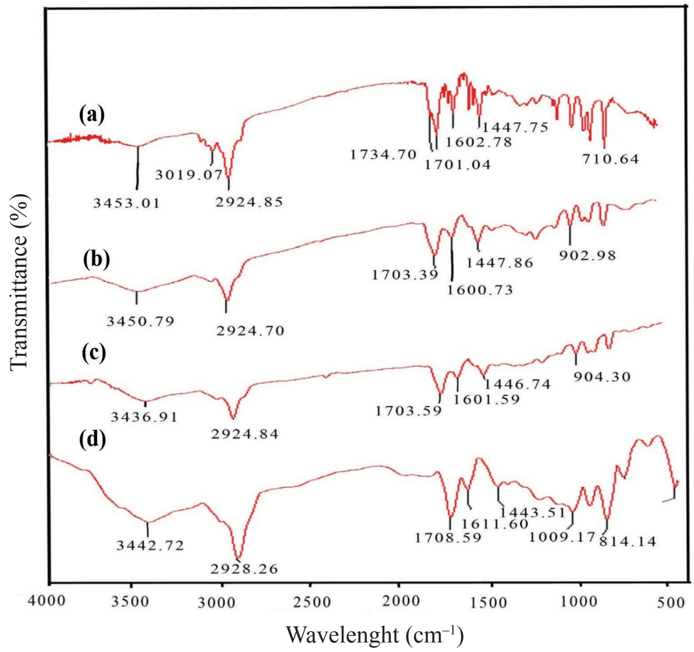


Figure 2: FTIR spectra of (a) MW80, (b) MW100, (c) MW110, and (d) MW120

The C=O stretching in the MAA shifted upfield, indicating that the production of crosslinked polymer and rigid networks increased with temperature. Therefore, the restricted movement of the C=O group leads to higher vibrational frequencies and causes blue shifting (Larkin, 2011).

Particle Morphology and Size of MIP

Figure 3 shows the morphology of MIP using FESEM spectroscopy. All MIPs prepared via MW irradiation showed smaller agglomerate spherical particles compared with MIP-C. The spherical particles were obtained using precipitation polymerisation in acetonitrile (Mohebali *et al.*, 2021). The morphological characterisation of all MIPs synthesised via MW showed distinct particle size reduction compared with the conventionally prepared MIP [Figure 3 (a)]. The average particle size of all MIPs was measured using ImageJ with 35 sample sizes (Figure 4). MW120 produced micro size polymer with the smallest particle sizes of 1.00 µm ± 0.24 µm while MW100, MW110, and MW80 gave decreasing polydisperse microparticle sizes of 1.25 µm ± 0.22 µm, 1.24 µm ± 0.17 µm, and 1.14 µm ± 0.25 µm, respectively (Shahar *et al.*, 2016). Rapid polymerisation via MW contributed to particle nucleation and growth during polymerisation, resulting in smaller particle sizes of the polymers (Burton & Goodwin, 2010). Although MW120 has a smaller particle size than MW110, the morphology of MW120 showed high polydispersity and agglomerated particles compared with MW110. The inhomogeneous distribution of binding sites in the agglomerated polymer particles results in reduced binding affinity (Zhao, 2022) and smaller surface area, thus, inducing fewer binding sites available to the template (Xia *et al.*, 2017).

The Effect of Particle Size on the Rebinding Capacity

The performance of the prepared MIP was evaluated for their rebinding efficiency. Figure 5 shows the rebinding capacity of the prepared MIP via MW in comparison to MIP-C. MW110

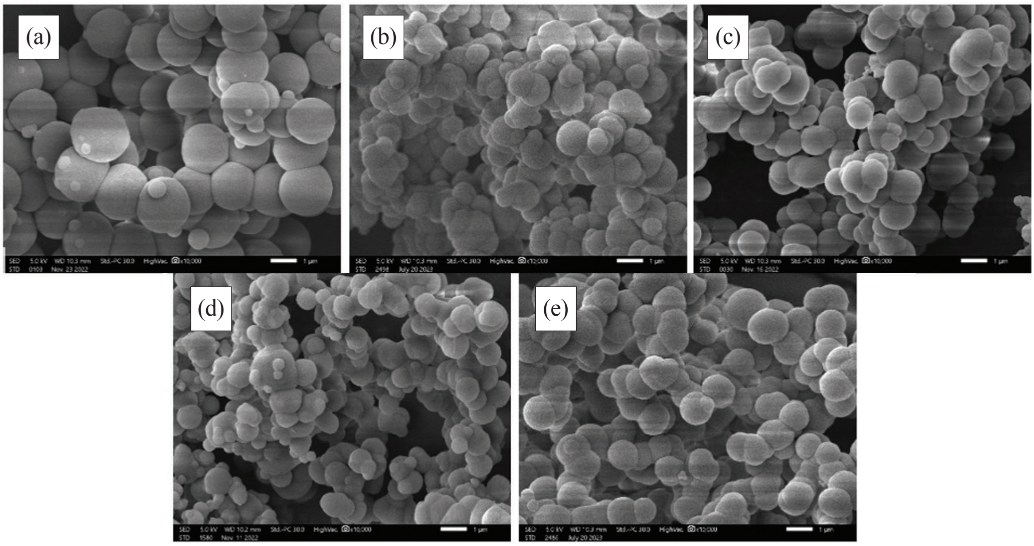


Figure 3: FESEM morphology of MIP at 10,000 x magnifications of (a) MIP-C, (b) MW80, (c) MW100, (d) MW110, and (e) MW120

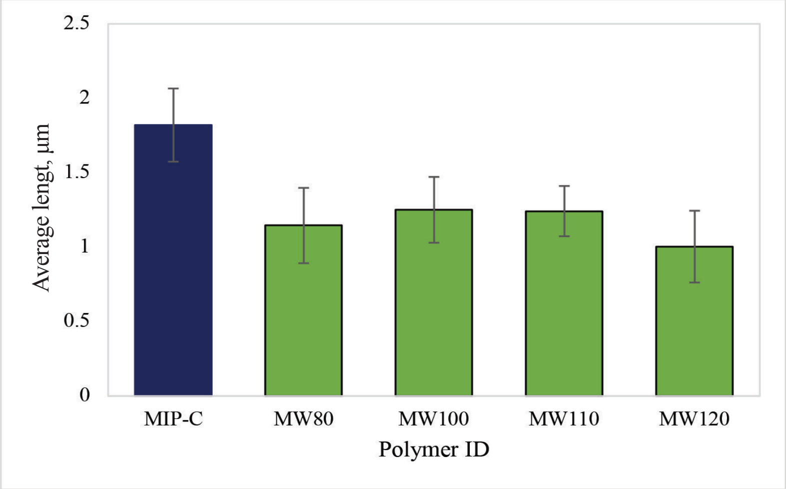


Figure 4: Average diameter of MIP. n = 35

showed the highest rebinding capacity at 7.27 mg/g ± 0.20 mg/g, compared with MW80 (5.33 mg/g ± 0.21 mg/g), MW100 (6.14 mg/g ± 0.27 mg/g), and MW120 (5.78 mg/g ± 0.38 mg/g). The size of MIP particles significantly influences the rebinding efficiency of MIP. Greater surface area-to-volume ratios that are frequently associated with smaller particle sizes might result in more binding sites and better access to the binding locations, which in turn can affect the rebinding capacity (Gkika *et al*., 2024). High rebinding capacity at 172 µmol/g was also observed in a similar study using MW in the preparation of MIP with particle sizes ranging from 2 µm to 5 µm (Khunsa-Ngiem *et al*., 2018). The low particle polydispersity of MW110 as compared to other MIPs has allowed more access to the template into the binding sites, resulting in higher rebinding capacity (Xia *et al*., 2017).

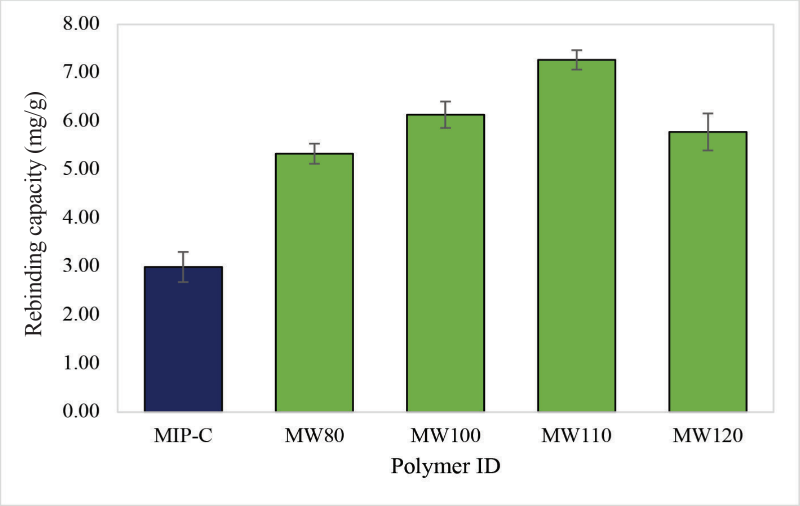


Figure 5: Rebinding capacity of MW MIPs compared to MIP-C

Conclusions

This research suggests that there is a significant advancement in the production of MIP via MW-assisted synthesis. Polymerisation via MW irradiation has produced a smaller particle size and high rebinding capacity compared with conventional polymerisation. Rapid polymerisation of MW110 has resulted in low particle polydispersity. Additionally, the high surface area-to-volume ratio of the particles has led to a high rebinding capacity. Regardless of the smaller particle size, MW120 demonstrated high particle polydispersity which in turn produced low rebinding capacity. The direct comparison with conventional thermal heating serves as a benchmark, highlighting the clear advantages of MW-assisted synthesis in achieving high rebinding capacity. This comparative analysis has positioned MW-assisted synthesis as a transformative methodology in the realm of MIP synthesis.

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Conflict of Interest Statement

The authors declare that they have no conflict of interest.

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