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Bamboo Activated Carbon-Magnetite as An Adsorbent for the Dispersive Micro-Solid Phase Extraction of Methyl Paraben and Hydroquinone in Skin Care Sample

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Abstract
This study investigated a newly synthesized bamboo activated carbon-magnetite (BAC-M) as an adsorbent for dispersive micro solid phase extraction (D-μ-SPE) of methyl paraben and hydroquinone paraben in a skin care sample. The synthesized adsorbent was characterized by Fourier Transform Infrared Spectroscopy (FTIR) and Field Emission Scanning Electron Microscopy (FESEM). Quantification of methyl paraben and hydroquinone was conducted using Gas Chromatography-Flame Ionization Detector (GC-FID). A few parameters of D-μ-SPE were optimized and evaluated, including sample pH, mass of adsorbent, extraction time, and volume of desorption solvent. The optimum conditions for this method were a sample pH of 5, 40 mg of adsorbent, a 3 min extraction time, and 70 μL of methanol as the desorption solvent. The optimized method showed good linearity, with R^2 values of 0.9963 and 0.9978 for methyl paraben and hydroquinone, respectively. In addition, the limit of detection (LOD) values were 2.60 mg/L and 2.97 mg/L for methyl paraben and hydroquinone. As for recoveries, both analytes showed satisfactory recoveries: 86.5% for methyl paraben and 117% for hydroquinone, with relative standard deviations (RSDs) < 10%. In the real sample analysis, both analytes were detected: methyl paraben at 2.7 mg/L and hydroquinone at 4.56 mg/L, with the latter still detected in the skin care sample despite its use being restricted by the Ministry of Health Malaysia.

Keywords Bamboo activated carbon-magnetite adsorbent, dispersive micro-solid phase extraction, paraben, hydroquinone, skin care

1.0 INTRODUCTION

With growing attention to personal beauty, a vast range of cosmetic products is being invented and commercialized worldwide nowadays [1]. However, the ingredients such as methyl paraben, hydroquinone, butylated hydroxytoluene, imidazolidinyl urea, etc., inside the product have raised concerns among consumers to ensure it does not contain any harmful components which could bring adverse effects to body health [2]. Methyl paraben and hydroquinone are two controversial ingredients widely used in cosmetic products due to their strong antimicrobial properties and whitening effects, respectively [3,4]. Parabens are a group of p-hydroxybenzoic acid (PHBA) esters that have served as preservatives for over 70 years in cosmetic products, consisting of methyl paraben, ethyl paraben, propyl paraben, isopropylparaben, butylparaben, isobutylparaben, and benzylparaben [5]. Due to its broad antimicrobial spectrum, good stability, and low volatility, this substance is extensively used to control the growth of bacteria to ensure a good condition of the product [3].

Methyl paraben is reported to be non-carcinogenic to humans, and it may be safely used at maximum authorized concentrations of 0.4% as acid for single ester and 0.8% as acid for a mixture of esters according to the Guidelines for Control of Cosmetic Products in Malaysia [6]. However, there was a study showing that methyl paraben possesses oestrogenic activity and how it is related to breast cancer [7]. For instance, there is a report that found that paraben was detected in 99% of breast cancer tissue samples, whereby in 60% of the samples, all five esters were measured [8]. Methyl paraben is the second highest with 16.6 ng per gram of tissues, with n-propyl paraben that comes first [8]. Therefore, it is unreasonable not to pay attention to the possible connection in the process of carcinogenesis [9].

Hydroquinone is an aromatic compound which is the derivative of benzene. It is well-known as a depigmentation agent in the cosmetic world and has been widely used as a skin-toning cream [10]. Hydroquinone is effective in decreasing the facial tone and inhibiting the formation of melanin in the skin [11]. In all parts of the world, especially in low and middle-income countries, the production and marketing of skin bleaching products has become a multi-billion-dollar industry [12]. This phenomenon is indeed worrisome, as there were many reports claiming that hydroquinone might bring adverse effects to health [10,12,13]. Skin bleaching may lead to dermatologic effects such as skin thinning, skin lesions, eczema, acne, and exogenous ochronosis (blue-black pigmentation formation) [13]. The use of hydroquinone in cosmetic products has been banned by the Health Ministry of Malaysia, as this chemical is only allowed to be used in the preparation of artificial nails and for professional use only [14]. The presence of this ingredient in cosmetic products may cause hypersensitivity of the skin, which lessens the protection from UV rays that can induce skin cancer [10].

Generally, the removal of interference before instrumental analysis is required, in which sample cleanup and pre-concentration of trace analytes from complex matrices are required [15]. Solid phase extraction (SPE) is said to be the most common and advantageous technique for sample preparation due to its straightforward and high selectiveness, but it has certain drawbacks such as huge secondary wastes, loss of solvent and a lengthy procedure [16]. These have led to the usage of miniaturized SPE, such as the dispersive micro solid extraction technique (D-μ-SPE), which can further improve the traditional SPE techniques [17]. D-μ-SPE is expected to be suitable for the analysis of small amounts of analyte due to small sorbent usage and lower consumption of organic solvent [18]. Additionally, the use of magnetite adsorbent for D-μ-SPE has remarkable effects, such as uncomplicated separation, simple operation conditions, and a simple manipulation process [19]. Magnetic separation technique has provided a new prospect in the evolution of adsorbent technologies, whereby it can be cost-effective, with extra efficiency as compared to the conventional ones [20]. Noteworthy, the selection of the adsorbent material is crucial to ensure a good extraction process efficiency. Several types of adsorbents, such as graphene oxide and surfactant, were invented for the extraction of cosmetic product ingredients [21,22]. Graphene oxide offered excellent extraction efficiency for aromatic compounds like methyl parabens and hydroquinone through strong π-π interactions with its large surface area [21]. However, this adsorbent was relatively expensive due to the high production cost, potentially suffering from aggregation issues with matrix interferences and deformation when exposed to sunlight [23-25]. Besides, surfactant-based adsorbents are relatively cost-effective and highlighted for their fast extraction performance [22]. Nonetheless, the smaller total surface area limited its adsorption capacity, resulting in poor extraction performance [26]. As an alternative, activated carbon is said to be a powerful adsorbent which has great application due to its high surface area and porous features, cost-effectiveness and high stability in diverse conditions [27]. Bamboo, a lignocellulosic biomass primarily composed of hemicellulose, cellulose and lignin, is used as activated carbon due to the high presence of carbon and fibrovascular bundles, which control the formation of activated carbon by acid treatment, forming a porous carbonaceous structure [28,29]. The magnetite (Fe_3O_4) is easily embedded in the bamboo activated carbon (BAC) as it exhibits low toxicity, high chemical stability, high adsorption capacity and promotes a simple preparation process [20]. Magnetite, which contains both Fe^{3+} and Fe^{2+} ions, is ferrimagnetic, which gives the magnetite a magnetic property. Together, the bamboo activated carbon-magnetite (BAC-M) can be used as an alternative to a cheap adsorbent of choice. Additionally, the interaction of the analyte with the adsorbent can be in the form of cationic exchange, polarity or π-π interaction [30]. One of the benefits of selecting bamboo as the precursor is that it is a renewable source that has a fast-growing rate [31].

After all, there was no report found on the use of BAC-M adsorbent to extract methyl paraben and hydroquinone, and yet it was proposed for the first time. Thus, the great features that bamboo possesses could be a great use for synthesizing activated carbon impregnated with magnetite as an adsorbent for the analysis of methyl paraben and hydroquinone in cosmetic products.

2.0 EXPERIMENTAL

2.1 Chemicals and Instrumentation

Iron (III) chloride hexahydrate ($FeCl_3.6H_2O$) was purchased from QReC (Rawang, Malaysia) and iron (II) sulphate heptahydrate ($FeSO_4.7H_2O$) was purchased from Systerm. Both were used in the preparation of BAC-M. Potassium nitrate (KNO_3) and sodium hydroxide (NaOH) were purchased from Merck (Darmstadt, Germany), and nitric acid (HNO_3) was used in the pH of zero-point charge (pH_{zpc}) study. Methyl paraben was purchased from Supelco (Bellefonte, PA, USA), and Hydroquinone was purchased from Merck (Darmstadt, Germany). They were prepared and used as stock solutions at a 1000 ppm concentration. Methanol was purchased from Merck (Darmstadt, Germany) and was used as a solvent. The quantification of methyl paraben and hydroquinone was conducted using gas chromatography-flame ionization detector (GC-FID; Agilent 6890N, USA) equipped with an HP-5 column (30 m x 0.22 mm i.d., 0.2 μm film thickness) according to the method of Farajzadeh and his co-workers, with some amendments [32]. Helium (99.9999%) was used as the carrier gas at

1.3 mL/min. The column temperature was initially programmed at 130 °C for 1 min, ramped to 200°C at 10 °C/min, and subsequently held at 200 °C for 2 min. The sample was injected in splitless mode with a purge time of 45 s. The injection port and flame ionization detector (FID) temperatures were set at 300 °C and 310 °C, respectively. The flow rate of hydrogen gas, which was generated via a hydrogen generator and air for FID, was fixed at 30 mL/min and 400 mL/min, respectively. The chemical composition of the BAC, BAC-M and Fe_3O_4 was characterized via Fourier transform infrared spectroscopy (FTIR; Perkin Elmer 1600, USA), and the samples were prepared using the potassium bromide (KBr) pellet technique with a 1:100 ratio of sample to KBr powder. The morphology of the synthesized adsorbent was investigated with Field Emission Scanning Electron Microscopy (FESEM; JEOL JSM-6701F, Japan).

2.2 Preparation of Bamboo Activated Carbon-Magnetite

Raw materials of bamboo stems (*Schizostachyum brachycladum*) were used as the precursor. Firstly, the bamboo was cut into 1 cm in size before being washed with distilled water and dried in the universal oven at 100°C for 24 hours. Then, 10 g of the dried bamboo shoots were soaked for another 24 hours in a 5:1 ratio with 55 wt. % H_3PO_4 at room temperature to ensure the phosphoric acid enters the interior of the bamboo material [33]. The excess acid was removed before drying the samples in the oven overnight at 80°C. After that, the sample was placed in the furnace and heated to 550°C and held for two hours. Later, the product was washed with distilled water to remove residual acid by using a centrifuge until the pH reached near 7.0. The product was dried again in the oven for 24 hours at 100°C, and the bamboo activated carbon obtained was ground and sieved into fine powder. The synthesized BAC was used for the preparation of BAC-M. Firstly, a 1:2 ratio of $FeSO_4$:$FeCl_3$, in which 0.7 g $FeCl_3$ and 1.4 g of $FeSO_4$ were weighed and mixed together in 100 mL of distilled water. The mixture was made homogeneous by stirring at 700 rpm without heat, and later the BAC was added to it. After 15 minutes, the mixture was added with another 100 mL of distilled water and heated in an oil bath at 60°C with the same stirring rate for 2 hours. After 2 hours, 10 M of NaOH was added dropwise until the pH reached 10. Here, the co-precipitation process occurred. The reaction was left for another hour. The mixture was cooled down and left until the precipitate settled at the bottom before being filtered. The magnetite was washed with ethanol to get the pH around 7. The product of BAC-M was vacuumed and dried overnight at 50°C in an oven.

2.2.3 Study on BAC-M Surface Charge

The BAC-M surface charge was investigated via pH_{zpc} by salt-addition method with 6 different pH values (pH 1, 2, 4, 6, 8 and 10). Initially, 0.1 M KNO_3 solution (40 mL) was poured into 5 different conical flasks (250 mL). The pH of the solution was adjusted using both NaOH and HNO_3 solutions (1 M). Subsequently, the BAC-M adsorbent (0.1 g) was added to each conical flask, and all of them were agitated using an orbital shaker with medium speed at room temperature overnight. The final pH value of the suspension (pH_f) was recorded on the next day. The change of the pH (ΔpH) was calculated using the pH_f and the initially adjusted pH value (pH_i) as summarised in Equation 1. The pH_{zpc} was determined based on the graph of ΔpH versus pH_i.

$$\Delta pH = pH_f - pH_i \quad \text{(Equation 1)}$$

2.3 Sample Preparation

The stock solution and sample preparation method were adopted based on Farajzadeh and his co-workers' study [32]. The stock solution of methyl paraben and hydroquinone (1000 mg/L) was prepared by dissolving each standard (0.0100 g) with methanol (10 mL). The stock solutions were stored and refrigerated at 4°C. The series of working standard solutions was freshly prepared by appropriate dilution of the stock solution to the required concentration. Body lotion obtained from a local drugstore was used in the determination of hydroquinone and methyl paraben content. The sample (0.1 g) was weighed and transferred into a volumetric flask (10 mL). Methanol (8 mL) was added and heated at 40°C in a water bath and occasionally shaken until dissolved. The solution was allowed to cool and made up to the mark with methanol. It was then filtered using a membrane filter to have a clear solution. The solution was later subjected to the D-µ-SPE procedure after being spiked with standards of methyl paraben and hydroquinone at different concentrations.

2.4 Dispersive Micro Solid Phase Extraction Procedure

The D-µ-SPE procedure was adapted based on the review of Khezeli and Daneshfar [19] with some modifications. Firstly, the working standard solution (5 mg/L, 5 mL) was spiked into a centrifuge tube (50 mL) containing BAC-M adsorbent (40 mg). The mixture was vortexed for 2 min to facilitate the adsorption of the target analytes on the surface of the adsorbent. The adsorbent was then magnetically separated using an external magnet, whereas the supernatant solution was decanted. The adsorbent containing analytes was desorbed by using a desorption solvent (100 µL) under sonication for 5 min. After sonication, the adsorbent was separated once again by an external magnet. The supernatant was collected and injected into the GC-FID. The overall procedure of D-µ-SPE was depicted in Figure 1.

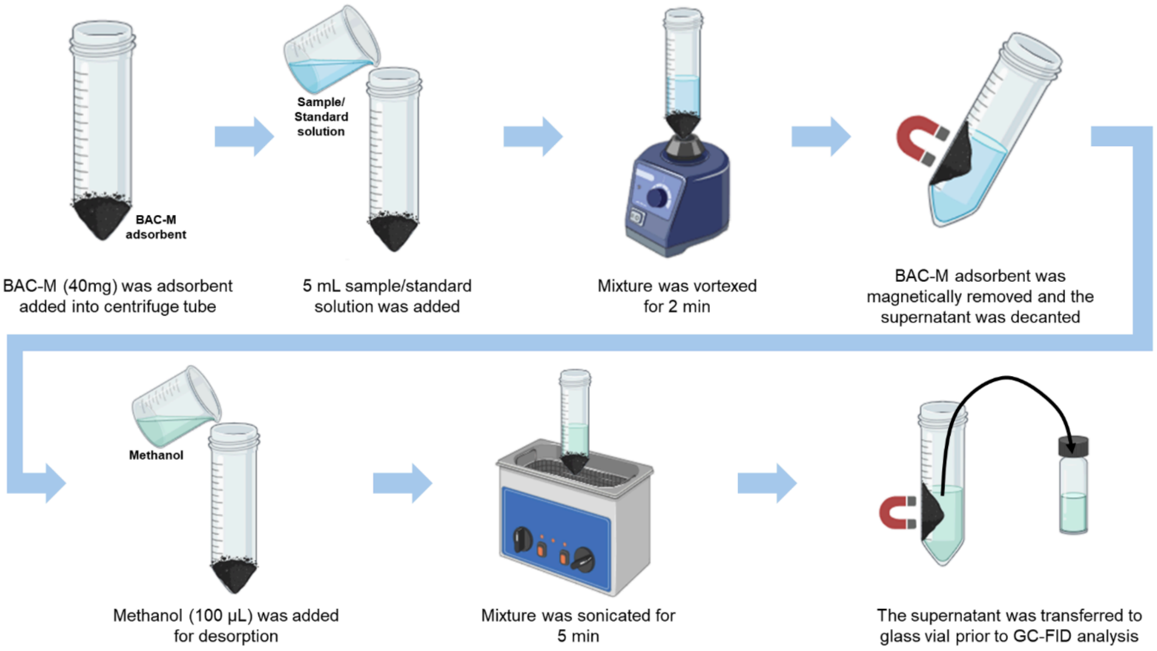

Figure 1 : Schematic diagram of D-µ-SPE

2.4.1 Method Optimization of D-µ-SPE procedure

Four key parameters of D-µ-SPE procedure, including the sample pH (2, 4, 6), mass adsorbent (20, 40, 60 and 80 mg), extraction time (0.5, 2.0, 3.0 and 5.0 min) and the desorption solvent volume (50, 70, 100 and 130 µL) were optimized based on the peak area obtained from GC-FID quantification to achieve optimal extraction performance for both methyl paraben and hydroquinone. The method optimization of each key parameter was performed using 5 mg L-1 mixture standard solutions of methyl paraben and hydroquinone in triplicate.

2.4.2 Method Validation of D-µ-SPE procedure

After all, the optimal parameters of D-µ-SPE were obtained, the method was further validated by evaluating the linearity, limit of detection (LOD), accuracy and intra-day precision. The linearity and LOD were evaluated via the standard addition calibration method by spiking the standard mixture solution (5, 10, 30 and 50 mg/L) in triplicate. Besides, the accuracy of the method was examined by spiking a certain concentration of the standard mixture solution in triplicate based on the relative recovery percentage. The intra-day precision of this method was conducted within a day, 3 times by spiking a certain concentration of standard mixture solution and evaluated based on the relative standard deviation percentage (%RSD).

3.0 RESULTS AND DISCUSSION

3.1 Characterization of Bamboo Activated Carbon Magnetite

FTIR analysis was conducted on the BAC, BAC-M and synthesized Fe_3O_4 to confirm the hybridization of magnetite onto BAC by the presence of the Fe-O bond in BAC-M. FTIR spectra in Figure 2 show a common vibration band at 3231 cm-1 and 3432 cm-1 for BAC and BAC-M, which belong to the O-H stretching. This was probably due to the presence of water during the synthesis. Another significant band that can be spotted at the range 1000-1300 cm$^{-1,}$ specifically absorption at 1184.64 cm^{-1} for BAC and 1018.07 cm^{-1} for BAC-M, have been assigned to C-O stretching in acids, alcohol, phenol ethers or esters group [34]. Another absorption which can be seen at 1635 cm^{-1}, 1627.89 cm^{-1} and 129.21 cm^{-1}, which belong to Fe3O4, BAC and BAC-M, is due to O-H bending. The most important attribute of these syntheses is the presence of the Fe-O bond in order to prove that Fe_3O_4 had been successfully hybridized into the BAC. The absorption at 592 cm^{-1} corresponded to the Fe-O bond in BAC-M, which can be further confirmed by comparing it with the synthesized Fe_3O_4. The Fe-O bond for Fe_3O_4 can be seen at 590 cm^{-1}.

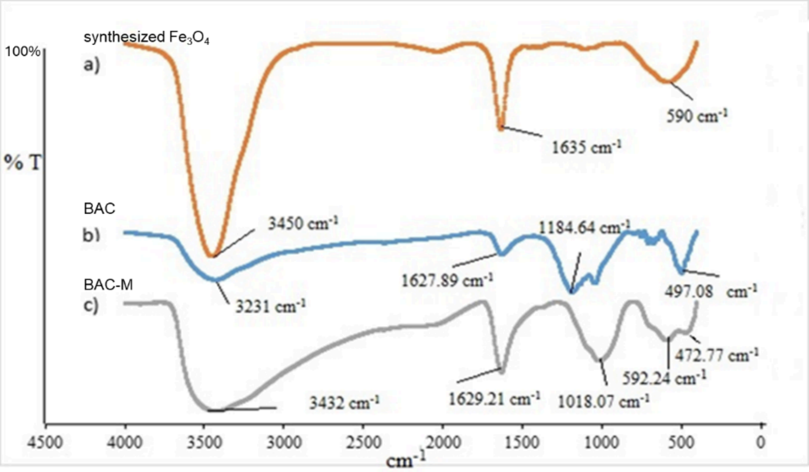

Figure 2: FTIR spectra of a) synthesized Fe_3O_4, b) BAC and c) BAC-M

FESEM was used to study the morphology of the material, in which the size and its shape can be observed, and the images of BAC, BAC-M and Fe_3O_4 were shown in Figure 3a-c. For the morphology of BAC, pores that can be observed were the structural 'channels' of cellulosic structure, but they are not pores. Those channels are formed, resulting from the elimination of organic and volatile compounds during the thermal decomposition and activation process [35]. The synthesized Fe_3O_4 surface appeared spherical due to coordination interaction between Fe atoms and 3d empty orbitals [36]. As for BAC-M, the surface was rougher compared to BAC, and this proves that the magnetite was successfully impregnated into the BAC.

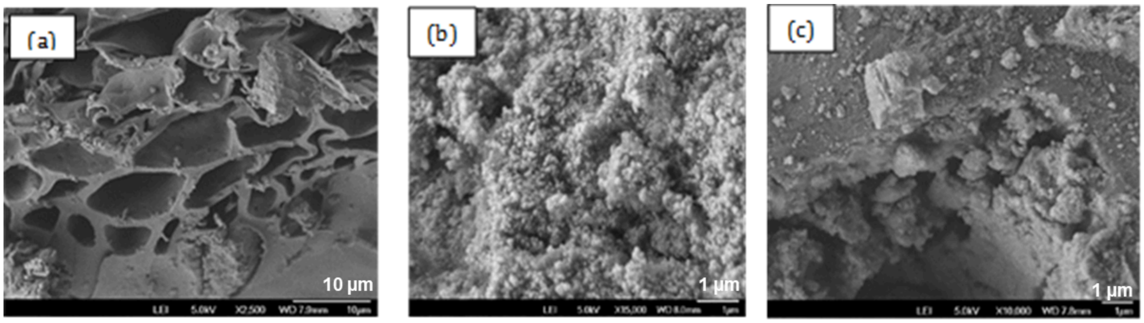

Figure 3: FESEM image of (a) BAC, (b) Fe_3O_4 and (c) BAC-M

pH_{zpc} by the salt addition method is a method that was used for the determination of the surface charge of the adsorbent. pH_{zpc} is defined as the surface of the material is neutral (zero surface charge) at particular pH value. pH of the surface charge is important as it influences the adsorption capability of the analytes onto the adsorbent. Principally, the surface charge of the material is positive when the pH of the sample is lower than pHzpc (pH sample<pHzpc), negatively charged when the pH of the sample is higher than pHzpc (pH sample > pHzpc) and neutral when the pH of the sample is equal to pH_{zpc} (pH sample = pH_{zpc}). Figure 4 shows the plotting of ΔpH against the pH_i. Based on the plotting, the ΔpH is zero at pH 4, meaning that the pH_{zpc} obtained was 4, suggesting that the surface of the BAC-M adsorbent was highly oxygenated [37]. By means, the BAC-M adsorbent was more negatively charged at alkaline conditions, which was similar to both weakly acidic analytes (methyl paraben and hydroquinone, with pKa values of 8.4 and 9.96, respectively). Therefore, the optimization range for the sample pH should be controlled near the pH_{zpc} to ensure the analytes and BAC-M adsorbents are deionized, which can promote a strong adsorption effect through hydrophobic interactions while reducing the electrostatic repulsion [38], enhanced the extraction performance of the D-µ-SPE.

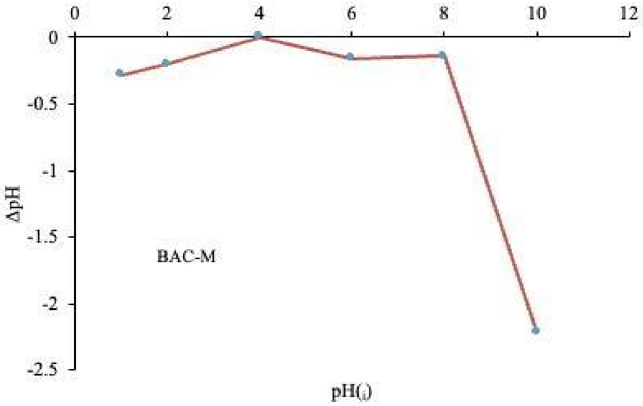

Figure 4: pH of zero-point charge for BAC-M

3.2 Optimization of Dispersive Micro Solid Phase Extraction Method

pH is the most crucial parameter in this optimization as it significantly governs the adsorption capability of the analyte onto the adsorbent. As such, pH did influence the interaction in which the surface charge of the adsorbent and the charge of the analyte are important for a satisfying interaction between these two. The analyte's charge depends on its pH and pK_a, such that when pH < pK_a, the analyte will undergo protonation, and the analyte will exist in deionized form. When pH >pK_a, deprotonation occurs and both compounds are in the form of anionic. pK_a for hydroquinone and methyl paraben are 9.96 and 8.4. Based on the results shown in Figure 5(a), pH 4 showed the highest peak area, followed by pH 2 and pH 6, but for hydroquinone, the highest peak area was at pH 6. Even though hydroquinone was at an advantage at this pH, the peak area for methyl paraben is very low. Hence, hydroquinone at pH 6 was compromised, and pH 4, since the peak area for both methyl paraben and hydroquinone was high for both. Since pH 4 was favoured, the interaction between analyte and adsorbent was probably due to hydrophobic and π-π interaction [39]. On the other hand, pH 2 and pH 6 showed low peak area as compared to pH 4. This is due to the reason that both compounds were in deionized form at pH<$pK_{a,}$ and the surface adsorbent were in charged form at pH 2 and pH 6. Hence, the interaction between the analyte and the adsorbent hardly occurred.

The influence of the mass adsorbent on the extraction efficiency was investigated by varying its value from 20 mg to 80 mg and keeping the other conditions constant. From the results obtained as shown in Figure 5(b), both methyl paraben and hydroquinone show an increasing trend from 20 mg to 40 mg. This shows that as the adsorbent mass increases, it provides more adsorption sites for the analyte. However, the peak area decreased after 60 mg and continually declined for 80 mg was probably due to the leaching of magnetite, hence the competition with the analyte [40]. Since the mass adsorbent 40 mg shows the highest peak area for this optimization, it was brought for the next optimization. The effect of extraction time on increasing the efficiency of the extraction was investigated. The extraction time was varied from 0.5 min to 4 min, and Figure 5(c) shows the outcome of this optimization. The result shows that 3 min was the best extraction time as compared to the others. The peak area increased as the extraction time increased, but at 5 min, there was an obvious decrement, which is probably due to back-extraction [30]. Hence, 3 minutes was chosen and taken for the next optimization. The volume of desorption solvent, which was methanol assisted by ultrasonication, was investigated, ranging from 50 µl to 130 µl. The peak area is escalated from 50 µl to 70 µl and further decreased from 100 µl to 130 µl. As shown in Figure 5(d), when less volume was being used, the peak area was high, and this is probably due to the analyte becoming more concentrated [41]. The decrease in extraction efficiency as the volume gets higher is the result of the dilution of analyte [41]. As a conclusion, the optimum extraction efficiency for this optimization was reached by using 70 µl of desorption solvent.

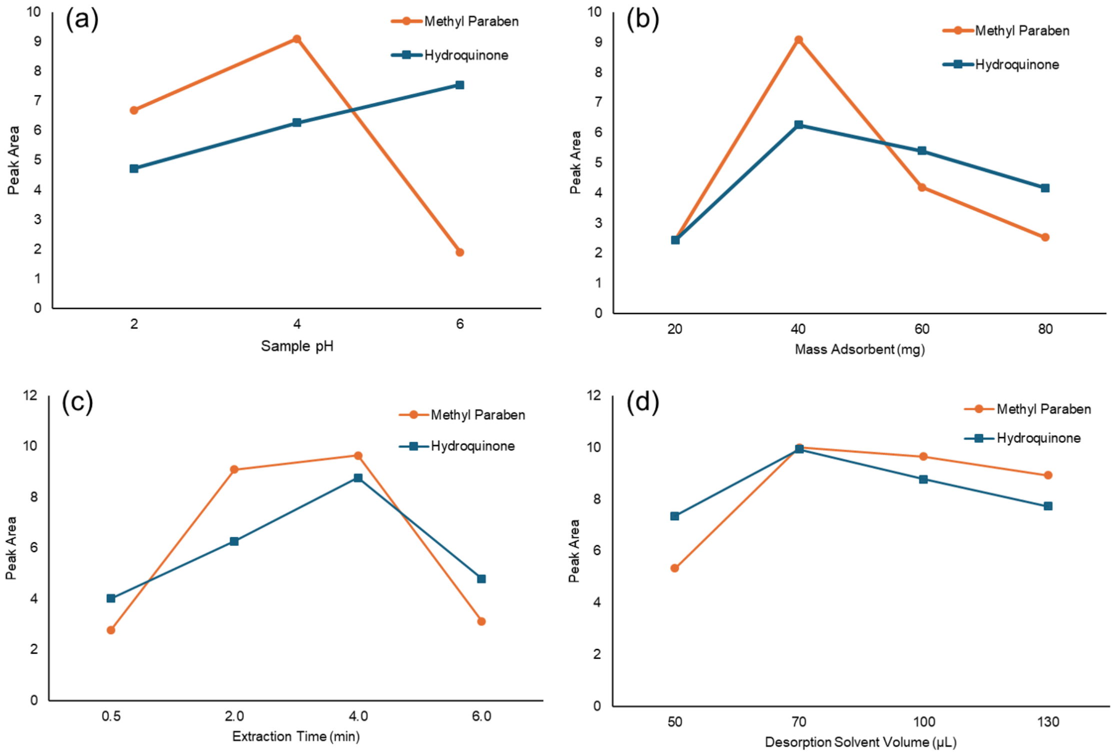

Figure 5 : Line chart of the method optimization for sample pH (other parameters: mass adsorbent=40 mg; extraction time=2 min; desorption solvent volume=100 µL), (b) mass adsorbent (other parameters: sample pH=4; extraction time=2 min; desorption solvent volume=100 µL), (c) extraction time (other parameters: sample pH=4; mass adsorbent=40 mg; desorption solvent volume=100 µL) and (d) desorption solvent volume (other parameters: sample pH=4; mass adsorbent=40 mg; extraction time=3 min; desorption solvent volume=100 µL)

3.3 Method Validation and the Analysis of a Real Sample

The method validation results of the BAC-M D-µ-SPE method were summarized in Table 1. The optimized method was validated under optimum conditions, under which a linear calibration plot was plotted in the range of 5-50 mg/L, which covered the concentration of methyl paraben and hydroquinone that were previously found in some cosmetic products, which were 28.7 mg/kg (equivalent to 41.00 mg/L) [42] and 20 mg/kg (equivalent to 28.57 mg/L) [43], respectively. It was observed that both analytes showed good linearity with satisfying R^2. The value of R^2 for methyl paraben was 0.9982; meanwhile, hydroquinone was 0.9978. As for the LOD, it was observed that the value for methyl paraben was 2.60 mg/L and hydroquinone was 2.97 mg/L. To assess the precision of this method, %RSD was used to evaluate its repeatability. The intra-day precision was done within a day, 3 times by examining the sample solution of a certain concentration. The %RSD for methyl paraben was 9.025% and hydroquinone was 5.460%. On the other hand, the accuracy was studied by determining the percent relative recovery. Based on the results gained, the percentage recovery for methyl paraben and hydroquinone was 86.5% and 117.0%. The BAC-M D-µ-SPE was applied to the analysis of a real skin care sample, which was obtained from a local drugstore. The sample was dissolved in methanol solvent, filtered and extracted prior to analysis. It was found that both methyl paraben and hydroquinone were detected in the skin care sample. The concentration of the methyl paraben detected was 2.70 mg/L, meanwhile, hydroquinone was 4.56 mg/L. The methyl paraben content, which exists as a single ester as listed in the ingredient, was still under the allowable amount, according to Guidelines for Control of Cosmetic Products in Malaysia, which was below 4 g/kg. Unfortunately, hydroquinone was detected, but the usage of this chemical has been banned by the Ministry of Health, Malaysia. Hence, the product is likely unsafe to use.

Table 1: Analytical performance of BAC-M D-µ-SPE in skin care sample

Analyte	Methyl Paraben	Hydroquinone
Linear Range	5-50 mg/L	5-50 mg/L
Linear Equation	y=2.0398x + 5.448	y=1.0575x + 4.4734
R^2	0.9963	0.9978
LOD (mg/L)	2.60	2.97
LOQ (mg/L)	7.88	9.00
Relative Recovery (%)	86.5	117
RSD (%)	9.02	5.46

4.0 CONCLUSION

From this study, BAC-M has been successfully synthesized for the extraction of methyl paraben and hydroquinone in skin care samples. This was proven by different characterizations of this adsorbent through FTIR analysis and FESEM, which can confirm the hybridization and the attachment of magnetite on the BAC. Besides, the optimum conditions for the D-µ-SPE method were achieved from the influence of several optimized parameters, which were a sample mixture of pH 4, 40 g mass adsorbent, 3 min extraction time and 70 µl volume of desorption solvent. Method validation was also shown to satisfy a correlation coefficient, LOD, relative recovery and repeatability, suggesting that the BAC-M as the adsorbent of the D-µ-SPE method provided efficient extraction and supported trace-level quantification of methyl paraben and hydroquinone in cosmetic samples.

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