Microencapsulation of bioactive volatile compounds from MD2 pineapple peel Extract using spray-drying and foam-mat drying

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ABSTRACT

Pineapple peel extracts constitute bioactive compounds that degrade due to light, oxidation, and moisture. This study aimed to encapsulate cellulase-treated MD2 pineapple peel extracts ensuring stability of bioactive compounds-rich extracts in the form of powders. Two drying methods were employed including spray-drying (150 °C) and foam-mat drying (60 °C) using maltodextrin (5 % v/v and 10 % v/v) and gum Arabic (GA) (5 % v/v and 10 % v/v) as carrier agents. Spray-drying for all microencapsulated powders exhibited high-quality powder with lower water activity (0.35–0.44), particle size diameters (D_{50}) ranging from 19.7 to 53.71 μm and higher solubility index (75.42–98.53 %) in comparison to foam-mat drying. Spray-drying using 10 % GA exhibited the highest encapsulation efficiency (EE%) of above 99 % and further extraction of different phenolic fractions is shown to be the most efficient encapsulation of insoluble-bound phenolic (IBP). Bioactive volatile compounds comprised of 2-Methoxy-4-vinylphenol and Phenol, 2,4-bis(1,1-dimethylethyl) in the microcapsules of 10 % GA. Cellulase-treated MD2 pineapple peel extract powder containing 10 % GA exhibited low in toxicity effect ($IC_{50} > 1000$ μg/mL) against NIH3T3 fibroblast cells. Microcapsule powder from bioactive-rich extracts of cellulase-treated MD2 pineapple peel has the potential to be used in functional foods, nutraceuticals, pharmaceuticals, and cosmetics ingredients.

1. Introduction

Pineapple (*Ananas cosmosus* L., Bromeliad family) has a therapeutic effect due to its richness in nutrients and bioactive compounds such as bromelain, polyphenols, fiber, vitamin C, and organic acids [1,2]. The fruit, which is often eaten fresh, can also be processed into delicacies such as juice, jam, preserves, jelly, and dried products [3]. Around 75 % of the pineapple fruit is a by-product of industrial processing, with the peel accounting for the largest proportion (15–60 %) and is underutilized for commercial [4–6]. Pineapple peel also contains important bioactive compounds such as ferulic acid [7], lactic acid, and succinic acid [8] as well as various aromatic substances [9]. Numerous research has emphasized the therapeutic potential of pineapple peel extracts, which have been demonstrated to be a moderate antimalarial agent in mice [10] and to protect against alcohol-induced oxidative damage in

the lungs and brain, as well as lipid peroxidation in rats [11,12]. However, the bioactive ingredients of pineapple peel extracts require appropriate processing, as incorporating the extracts directly into the products can reduce the effectiveness of the ingredients. Moreover, the stability of the extracts during storage between production and use is crucial, which can be achieved by microencapsulation [13,14]. Microencapsulation could protect the bioactive compounds from sensitivity to light, temperature, and oxygen [15].

High-quality powder characteristics produced through encapsulation should comprise microcapsules ranging from 1 μm to 100 μm [16], 3–800 μm [17], or 1 μm to less than 5000 μm [18]. Furthermore, characteristics of encapsulated microcapsules include convenient transportation and storage [19,20], lower particle size with increased water solubility [13,21], and reduced microbiological deterioration due to low water activity [20].

Spray drying is an efficient technique for preserving bioactive compounds. The drying technique increases natural active chemical compounds' bioavailability and solubility of low-soluble compounds, is low-cost, rapid, and continuous production and convenience in industrial settings [22–25]. Microencapsulation by spray-drying was compared with freeze-drying on encapsulation of phenolic compounds from ciriguela peel [24]. Meanwhile, anthocyanins extracted from roselle were encapsulated to study the effect of different carrier agents on their physicochemical properties using either spray-drying or freeze-drying methods [15]. Moreover, several studies have demonstrated the possible application of spray drying on agricultural waste phenolic compounds-rich extract, including that from lemon waste [26], red pitaya peel [27], Euterpe edulis co-product [28], and cocoa shells [29].

Another promising drying technique in microencapsulation is foam-mat drying. Stable foams are created from whipped liquid or semi-liquid foods with the addition of a foaming agent and/or stabilizing agent before drying [30]. To obtain high-quality characteristics of bioactive-rich powders, foam-mat drying should include processing at low temperatures (40–60 °C), foam thickness (0.2 and 1 cm), and egg white used as foaming agents leading to high retention of bioactive compounds [31]. Recent research focused on the total phenolics content, and antioxidant capacity of foam-mat dried turmeric extract [32]. In another study, watermelon rind powder was developed using foam mat drying in which maltodextrin as the wall material increased solubility provided with low moisture content [33].

Since pineapple peel extracts have potential as ingredients, the extracts' stability is crucial during processing, distribution, and storage. One emerging approach is using enzymes as an extraction medium to transform food waste into bioactive compounds, biodegradable plastics, sweeteners, functional sugars, biofuels, and many more [34]. Nevertheless, the challenges remain for pineapple peel extracts in liquid form as it susceptible to harsh environments such as light, moisture, oxygen, and humidity which may lose the beneficial components of its bioactive compounds.

Therefore, this study aimed to evaluate the effectiveness of enzyme treatment on MD2 pineapple peel extract for microencapsulation method by either spray-drying or foam-mat drying in producing high-quality bioactive-rich powders. The specific aim of this study was to evaluate the physical and functional characteristics of microencapsulated pineapple peel extracts using different carrier agents and drying techniques. In addition, the cytotoxicity activity of the extract powders was evaluated to determine their potential as an ingredient in pharmaceuticals, nutraceuticals, and cosmetics.

2. Materials and methods

2.1. Material and reagent

Analytical grade (AR) solvents and reagents were used. Generally registered as safe (GRAS) cellulase (Celluclast 1.5L, Novozymes, Bagsværd, Denmark). Food chemical codex (FCC) grade and generally registered as safe (GRAS) GA and maltodextrin (DE 10–15) were purchased from Markaids (Malaysia) Sdn Bhd and Scienfield Expertise PLT (Malaysia). The NIH3T3 (mouse embryonic fibroblast) cell line was obtained from the American Type Culture Collection (ATCC, Manassas, VA, USA). RPMI-1640 medium with L-glutamine (Nacalai Tesque, Kyoto, Japan) was used to cultivate NIH3T3 cells. 10 % fetal bovine serum (FBS) (Cytiva, Freiburg, Germany) and 1 % penicillin-streptomycin (GeneDireX, Taiwan, China). Cells were incubated at 37 °C with 5 % CO_2 and 95 % air.

2.1.1. MD2 pineapple peel collection
MD-2 pineapple peel collected from the Department of Agriculture in Serdang, Selangor, Malaysia were based on the maturity characteristics of Index 2, and 25 % yellow peel base as performed by Nordin et al. [35]. In brief, pineapple peels were placed in a 1 kg woven PP bag. After being brought to the laboratory, pineapple peels were cleansed to get rid of any debris. The samples were sliced into 1 cm × 1 cm x 1 cm pieces for further analysis.

2.2. Preparation of cellulase-treated MD2 pineapple peel extracts

The cellulase treatment was performed on MD-2 pineapple peel extracts following Nordin et al. [35] with modifications on the extraction temperature and time. In brief, small strips of peels (1 cm × 1 cm x 1 cm) blanched in a water bath (Model WNB 14, Memmert GmbH + Co. KG, Schwabach, Germany) at 95 °C ± 5 °C for 5 min. Then cooled down at a room temperature of 22 °C ± 1 °C. Next, 75 g of samples were homogenized (Model BL3071, Tefal (400W) Blendforce Blender) for 60 s, and 300 mL distilled water was added in the ratio of 1:4 (g/mL). Cellulase (1.5 % (v/v)) was added to the homogenized peel mixture following the parameter condition (48 °C ± 0.2 and 132 min) optimized in a preliminary experiment. The cellulase treatment was carried out in a covered thermostatic water bath (Model WNB 14, Memmert GmbH + Co. KG, Schwabach, Germany) at 48 °C ± 0.2 with agitation (150 rpm) in the dark for 132 min. Enzyme hydrolysis was halted at 90 °C ± 5 for 5 min. The samples were cooled down for 5 min at room temperature (22 °C ± 1 °C).

2.3. Microencapsulation by spray-drying process

Preparation of the spray-dried cellulase-treated MD2 pineapple peel powder extract was carried out according to Lourenco et al. [13] with modification on the extraction method. The encapsulation of cellulase-treated MD2 pineapple peel extract was done by mixing a carrier agent with the extracts before spray drying. The effect of different carrier agents with concentrations of 5 % (v/v) and 10 % (v/v) of maltodextrin (DE 10–15) and GA was studied. The concentrations of gum Arabic and maltodextrin both at 5 % and 10 % were based on previous literature research. In spray-drying, the established method by Luorenco et al. [13] used a 5 % (w/w) carrier agent to encapsulate the peel extracts provided with high-quality powder characteristics. Furthermore, cellulase-treated MD2 pineapple peel extracts have a low total soluble solids (2.94° Brix) which only requires low concentrations of carrier agents to be used. Therefore, to ensure high-quality powder characteristics and cost-effectiveness, only 5 % and 10 % concentrations were chosen in the encapsulation process using spray-drying and foam-mat drying methods.

Maltodextrin and GA were heated at temperatures of 40 °C ± 5 before the addition of the extract. Then, a mixture of carrier agents and cellulase-treated MD2 pineapple peel extract was performed under magnetic stirring for 30 min at 1000 rpm as feed solution (Cimarec Stir Hotplate, Thermo Fisher Scientific, Waltham, Massachusetts, US). Consequently, extracts of feed solution were subjected to a spray dryer (Mobile Minor® R&D GEA Niro, Düsseldorf, Germany), with an atomizer nozzle or co-current flow regime, equipped with a 0.7 mm diameter nozzle. The pump feed flow rate and atomizing pressure of the compressed air were set at 14 rpm and 1.8 bar, respectively. Feed solutions were dried at inlet and outlet air temperatures of 150 °C and 90 °C, respectively. The dried powders collected were sealed in an aluminum pouch and stored at an ambient temperature of 22 °C ± 1.

2.4. Microencapsulation by foam-mat drying process

The foam-mat drying was carried out following Gao et al. [36] with modifications in the drying process. Two foaming agents: 0.5g/100g cellulose methoxyl carbonate (CMC) and 8g/100g egg albumin (EA) (Modernist pastry). Meanwhile, carrier agents used were maltodextrin (DE 10–15) and GA at 5 % (v/v) and 10 % (v/v), respectively. Foaming agent solutions of 0.5 g CMC, 8 g EA, and 5 % and 10 % of each maltodextrin and GA powders were diluted in 100 mL distilled water and heated at 70 °C ± 5 for 30 min in a water bath (Model WNB 14,

Memmert GmbH + Co. KG, Schwabach, Germany). Then, the mixtures of CMC, EA, and foam stabilizers were gradually added to 125 mL of the cellulase-treated MD2 pineapple peel extracts. The extracts and foaming agent mixtures were whipped using a Food processor (Model MK-5087M, Panasonic Sdn Bhd, Malaysia) for 10 min until a foamed structure was formed. The foamed sample was layered on a tray covered with baking paper with a height of 3 mm. Samples were subjected to an air flow oven at a temperature of 60 °C ± 1 for 12 h until the moisture content reached 6 % and below.

Spray-drying and foam-mat drying were chosen as both techniques are among established industrial hot air drying methods for encapsulation of fruits into powder ingredients due to their high encapsulation efficiency, high solubility of powders, and ability to retain bioactive and antioxidant compounds at low cost. Previous research had highlighted the significant use of spray-drying in encapsulating pomegranate peel water extract which could retain its antioxidant compound with a high solubility index [37]. An important emerging field in foam-mat drying is the encapsulation process at low temperatures (40–60 °C), foam thickness (0.2 and 1 cm), and use of egg white as a foaming agent which could result in high retention of bioactive compounds [31]. The specific temperatures for this study were selected based on previous research. For spray-drying, the lowest temperature of 150 °C was obtained from the method of Luorenco et al. [13]. The selected temperature was chosen due to the higher antioxidant compound retained in the microcapsules after spray-drying. Meanwhile, the temperature and drying times for foam-mat drying were selected from the preliminary experiments in which using temperature at 60 °C for 12 h provided moisture content of 6 % and below as compared to 50 °C which the sample requires extensive duration for moisture content to reach 6 % and below.

2.5. Moisture content

Moisture content was performed using a Moisture Analyzer (Model MF50, A & D Moisture Balance, A & D Company Limited, Tokyo, Japan). About 2.0 g of cellulase-treated MD2 pineapple peel extract powder was weighed on the moisture balance and measurement was recorded in triplicates.

2.6. Water activity (a_w)

Measurements of water activity (a_w) in cellulase-treated MD2 pineapple peel extract powder were performed using a water activity meter (Aqualab 3 TE, Decagon Devices Inc., Pullman, Washington, US) of water activity ranging between 0.03 and 1.00. Measurements were done in triplicates.

2.7. Bulk density

The bulk density of cellulase-treated MD2 pineapple peel extract powder was calculated using Eq. (1) according to Yang et al. [37]. Approximately 5 mL of microcapsules were weighed in a 50 mL cylinder. The mass (g) and volume (cm³) of powders were recorded and calculated as *m/v* to obtain bulk density.

$$\text{Bulk density} \ (\text{g} / \text{cm}^3) = \frac{\text{Mass of powder (g)}}{\text{Volume (cm}^3)} \tag{1}$$

2.8. Tapped density

Tapped density was conducted by weighing 5 mL of microcapsules in a 50 mL cylinder. The cylinder was tapped 10 times until a constant weight was achieved. The constant mass (g) and volume (mL) were recorded and calculated as *m/v* using Eq. (2).

$$\text{Tapped density} \ (\text{g} / \text{mL}) = \frac{\text{Mass of powder (g)}}{\text{Volume (ml)}} \tag{2}$$

2.9. Water solubility index (WSI)

Measurement of the water solubility index (WSI) was done using Eq. (3) according to Nguyen et al. [15]. About 0.1 g microcapsules were diluted in 10 mL distilled water and stirred for 5 min on a hotplate (Cimarec Stir Hotplate, Thermo Fisher Scientific, Waltham, Massachusetts, US). Next, centrifugation of mixtures was conducted at 3000 rpm for 10 min. Subsequently, the supernatant was transferred onto a glass Petri dish and dried in an oven at a temperature of 150 °C ± 1. The WSI (%) is calculated as the mass of dried supernatant/mass of initial powder.

$$\text{WSI} \ (\%) = \frac{\text{Mass of dried supernatant}}{\text{Mass of initial powder sample}} \ \text{x} \ 100 \tag{3}$$

2.10. Flowability and cohesiveness

Flowability and cohesiveness of the cellulase-treated MD2 pineapple peel extract powder were calculated using Eqs. (4) and (5) according to the methods of Lourenco et al. [13] and calculated as $(\rho t- \rho b)/\rho t$ x 100 and $\rho t/\rho b$, respectively. The classification of CI (%) and HR of powders are based on Jinapong et al. [38] as shown in Table 1.

$$\text{CI} = \frac{(\rho t - \rho b)}{\rho t} \times 100 \tag{4}$$

$$\text{HR} = \frac{\rho t}{\rho b} \tag{5}$$

2.11. Particle size distribution

Measurements of particle size distribution were done according to Yang et al. [37] on the microcapsules of spray-dried and foam-mat dried cellulase-treated MD2 pineapple peel extract powder using a particle size analyzer (Mastersizer 2000 Scirocco Malvern Instruments Ltd., Worcestershire, UK). A laser diffraction technique was used by the dispersion module Sirocco 2000. The volume-weighted mean diameter was represented as diameter D [3,4]. The calculation of span value for particle size distribution was used following Yang et al. [37] as in Eq. (6).

$$\text{Span} = (d(0.9) - d(0.1))/d(0.5) \tag{6}$$

2.12. Total Phenolic content

The structure of microcapsules was disrupted following the evaluation of total phenolic content (TPC) by Ghandehari Yazdi et al. [39]. Microcapsules (200 mg) were added to a mixture of 2 mL ethanol/acetic acid/water (50:8:42) and vortexed for 1 min. Then, the mixture was centrifuged (EBA 20, Hettich Centrifuge, Föhrenstraße, Tuttlingen, Germany) at 6000 rpm for 5 min. The supernatant was filtered using a 0.45 μm pore size chromatography syringe filter. The TPC was quantified according to Hossain and Rahman [40]. After adding 0.2 mL of Folin-Ciocaltaeu reagent to a test tube containing a 200 μL extract, the tube was vortexed for 15 s. One mL of 15 % sodium carbonate solution

Table 1
Classification of flowability and cohesiveness of powders.

CI (%)	Flowability	HR	Cohesiveness
<15	Very good	<1.2	Low
15–20	Good	1.2–1.4	Intermediate
20–35	Fair	>1.4	High
35–45	Bad		
>45	Very bad		

was added to the mixture after 4 min, and incubated in the dark at room temperature for 2 h. Blank was prepared in the same manner without extracts. The absorbance of mixtures was measured by UV–Vis Spectrophotometer at 765 nm against a blank (GENESYS™ 10S, Thermo Scientific™, Waltham, MA, USA). The standard calibration curve was prepared in the same manner using Gallic acid standard solutions of 0, 25, 50, 100, 150, and 250 μg/mL, $R^2 = 0.9571$. The total phenolic content was expressed as mg of Gallic acid equivalents (GAE)/g of dry weight (DW) and calculated as in Eq. (7). Where, Total Phenol Content in mg/g, in GAE, C_1 = concentration of Gallic acid obtained from the standard curve in mg/mL, DF = Dilution Factor, v = volume of extract in mL, and m = the weight of the microcapsules in gram.

$$\text{Total phenolic content (TPC)} = C_1 \times DF \; X \; \frac{V}{m} \qquad (7)$$

2.13. Surface phenolic content

The surface phenolic compounds of microcapsules were done according to Yang et al. [37]. A 200 mg of microcapsule extract powder was added to 1 mL methanol and 1 mL ethanol (1:1 v/v) and vortexed for 1 min. Then, to obtain a clear filtrate a 0.45 μm pore size chromatography filter was used. Next, the concentration of surface phenolic compounds was determined according to the Folin-Ciocalteu method as described in the TPC section.

2.14. Encapsulation efficiency (EE%)

The encapsulation efficiency (%) was determined according to Yang et al. [37] calculated as in Eq. (8).

$$\text{EE (\%)} = \left(1 - \frac{\text{Surface phenolic content of encapsulated MP}}{\text{Total phenolic content of encapsulated MP}}\right) \times 100 \qquad (8)$$

2.15. Extraction of free, soluble-conjugate, and insoluble-bound phenolic

Extraction of free phenolic, soluble-conjugated phenolic (esterified and glycosylated) and insoluble bound phenolic was conducted according to a method by Wang et al. [41]; Arruda et al. [42]; and Yao et al. [43].

2.15.1. Free phenolic fraction

Extraction of free phenolics fraction was performed according to Wang et al. [41]. Spray-dried and foam-mat dried cellulase-treated MD2 pineapple peel extract powder of 0.5 g was diluted in 10 mL of 70 % methanol. The extraction was done in triplicate. Then, extract mixtures were homogenized in a water bath at 40 °C, 150 rpm, and for 1h. Subsequently, the obtained filtrate was extracted three times with 70 mL ethyl acetate through liquid-liquid extraction. Ethyl acetate extract was labeled as the organic phase, while the remaining extract was labeled as the aqueous phase. Consequently, ethyl acetate extract was redissolved in 5 mL of 50 % methanol and labeled as free phenolics. Meanwhile, aqueous phase extract was used for soluble-conjugate phenolics extraction.

2.15.2. Soluble-conjugated phenolic (esterified and glycosylated)

The extraction of esterified and glycosylated phenolics fractions was performed following Wang et al. [41] and Arruda et al. [42]. First, soluble-conjugated phenolics extraction was performed on the obtained aqueous extract from free phenolics. Then, the addition of extracts was done with 40 mL of 2 M NaOH for 4h at room temperature (23 °C ± 1 °C) and acidified with 12 M HCl at pH 2.0. Next, hydrolysate was extracted three times with 70 mL ethyl acetate through liquid-liquid extraction. Subsequently, the ethyl acetate phase was labeled as the organic extract, while the aqueous phase as the remainder extract. The organic extracts were evaporated under a rotary evaporator at 40 °C ± 1 °C until dry. Later, the obtained extract was redissolved in 5 mL of 50

% methanol (v/v). Glycosylated phenolics were extracted using the remainder of the aqueous phase extract. For further analysis, the extracts were stored at −20 °C ± 1 °C.

Aqueous phase extract was used in the extraction of glycosylated phenolics and was carried out according to a method performed by Arruda et al. [42]. Acid hydrolysis was done on the aqueous phase extract using 5 mL of 6 M HCl at 75 °C and 150 rpm for 60 min. Next, liquid-liquid extraction was performed thrice on the hydrolysate using ethyl acetate. Ethyl acetate extract was dried at 35 °C ± 1 °C using a rotary evaporator until the formation of crystal powder. Later, the obtained extracts were redissolved in 5 mL of 50 % methanol (v/v). For further analysis, the extracts were stored at −20 °C ± 1 °C.

2.15.3. Insoluble-bound phenolic

The extraction of insoluble-bound phenolics (IBP) was done according to Arruda et al. [42]. Residues obtained from free phenolic extraction were used for the IBP fraction. First, the residues were subjected to alkali hydrolysis using 50 mL of 2 M NaOH at room temperature (23 °C ± 1 °C) for 4h. Hydrolysate was acidified using 12 N HCl until pH 2.0 was obtained. Subsequently, mixtures were extracted thrice using 70 mL ethyl acetate through liquid-liquid extraction. The fractions of ethyl acetate were dried using a rotary evaporator at 35 °C ± 1 °C until the formation of crystal powder. Insoluble-bound phenolics extract was redissolved in 5 mL of 50 % methanol (v/v). For further analysis, the extracts were stored at −20 °C ± 1 °C.

2.16. DPPH free radical scavenging assay

The antioxidant activity of cellulase-treated MD2 pineapple peel extract powder was evaluated by 2,2-diphenyl-1-picryl-hydrazyl-hydrate (DPPH) free radical scavenging assay of Hossain and Rahman [40] with modifications on the sample preparation. Microcapsules (200 mg) were added to a mixture of 2 mL ethanol/acetic acid/water (50:8:42) and vortexed for 1 min. Then, the mixture was centrifuged (EBA 20, Hettich Centrifuge, Föhrenstraße, Tuttlingen, Germany) at 6000 rpm for 5 min. The supernatant was filtered using a 0.45 μm pore size chromatography syringe filter. Then, 800 μL of supernatant extract from different samples was pipetted into test tubes. Later, a 5 ml aliquot of ethanolic DPPH solution (0.1 mM) was added to each extract. The mixtures were vortexed and left to stand for 20 min at 25 °C ± 1 °C. Ethanol served as a blank, and the control absorbance was prepared in the same method without the extract. The absorbance values were measured with a UV–Vis Spectrophotometer at 517 nm (GENESYS™ 10S, Thermo Scientific™, Waltham, MA, USA). DPPH free radical scavenging assay was calculated as in Eq. (9).

$$\text{Inhibition (\%)} = \frac{(\text{Control Absorbance-Sample Absorbance})}{\text{Control Absorbance}} \qquad (9)$$

2.17. Ferric reducing antioxidant power

The antioxidant activity of microencapsulated powders using a Ferric reducing antioxidant power (FRAP) assay was performed according to Lau et al. [44]; Benzie & Strain [45]. First, the preparation of the FRAP reagent consisted of using acetate buffer (300 mM, pH 3.6), diluted HCl (40 mM), TPTZ (10 mM), and ferric chloride solution (20 mM). A mixture of the working FRAP reagent was in the ratio of 10:1:1 (acetate buffer: TPTZ solution: ferric chloride solution). Briefly, 3.0 mL FRAP reagent was added to 100 μl of standard, blank (solvent to extract sample) and samples. Then, incubation of the reaction mixture was done at 37 °C for 4 min. Consequently, absorbance was measured using a UV–Vis spectrometer at 593 nm (GENESYS™ 10S, Thermo Scientific™, Waltham, MA, USA). A standard curve was prepared using ferrous sulphate (100–1000 μmol). Results were expressed as mM Fe^{2+}/g DW.

2.18. GC-MS analysis

Qualitative screening of volatile compounds was performed according to Hernandez Escarcega et al. [46] and Kamaraj et al. [47]. First, an amount of 0.2 g microcapsules was extracted in a 10 mL methanol-water mixture (95 % v/v) for 60 min. Then, centrifugation was performed on filtrates at 1000 rpm for 10 min and further filtered through a chromatography filter pore size 0.45 μm (Reg. cellulose syringe filter, Titan 2). Next, each clear sample (1.0 μL) was injected into gas chromatography (Model 7890A, Agilent Technologies, Palo Alto, USA). GC-MS analysis was conducted using the Agilent Technologies GC systems 7890A model (Agilent Technologies, Santa Clara, CA, USA) equipped with an Agilent 5975 inert mass selective detector (MSD) to identify the compounds by using an HP-5MS column (30 m in length x 250 μm in diameter x 0.25 μm in thickness film). At a flow rate of 1 mL/min, pure helium gas (99.995 %) operated as the mobile phase. The electronic ionisation mechanism of the mass spectrometer detector employed high-energy electrons with a potential energy of 70 eV. The temperature was first adjusted to be between 70 and 150 °C, rising at a rate of 5 °C per min, and held for approximately 5 min. Then, the temperature rose at a rate of 15 °C each minute to 300 °C. A splitless mode injection of 1 μL of the extract was used. The percentage of chemical components in each powder extract was determined by observing the peak area that was generated in the chromatogram. Using a mass spectrum from GC-MS, the chemicals were identified, and their interpretation was based on a National Institute Standards and Technology (NIST) match against over 62,000 patterns. The sample components of the mass spectra were compared to those kept in the library and published in publications. The identity and confirmation of the chemical name, molecular formula, and percentage area were obtained.

2.19. Cytotoxicity (MTT assay)

The 3-(4,5-dimethylthizaol-2-yl)-2,5-diphenyl tetrazolium bromide (MTT) assay was performed according to Foo et al. [48] with modifications on cell type, incubation period, and amount of DMSO. Briefly, NIH3T3 cells (normal, non-tumorigenic cells) were trypsinized (trypsin-EDTA (1 ×), Cytiva, Freiburg, Germany) and seeded in 96-well flat-bottomed plates at a density of 5×10^4 cells/mL with 5000 cells per well in 100 μL of complete culture media (CCM), followed by incubation at 37 °C (5 % CO2 and 95 % air) for 24 h to allow cells attachment. The cells were then treated with either 5 % DMSO (ChemAR, Kielce, Poland) (served as a positive control), pineapple peel extract (62.5–1000 μg/mL) and further incubated for 72 h. Control cells were incubated with 100 % CCM. Following incubation, 20 μL of MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide), Solarbio, China) (5 mg/mL in phosphate buffer saline) was added into each well and the plate was incubated for 4 h. Following the aspiration of the excess MTT, 150 μL of DMSO was added to dissolve the formazan crystals that had formed. Consequently, absorbances were measured at 570 nm with a reference wavelength of 630 nm by using the Infinite F50 Absorbance Microplate Reader (Tecan Trading AG, Switzerland). Cell viability (%) was calculated as in Eq. (10).

$$Cell \ viability \ (\%) = \frac{Absorbance \ sample}{Absrobance \ control} \times 100\% \qquad (10)$$

2.20. Statistical analysis

Data were presented as mean values ± standard deviation. Analysis of variance (ANOVA) with Tukey's test was used to evaluate statistical significance with a 95 % significance level (p < 0.05) using Minitab software.

3. Results and discussion

3.1. Moisture content and water activity (a_w)

Moisture content and water activity (a_w) are important indicators of a powder product's quality [15]. Table 2 shows the moisture contents and water activity (a_w) of spray-drying and foam-mat drying of cellulase-treated MD-2 pineapple peel extracts using different carrier agents. The lowest moisture and water activity (a_w) was observed in the microcapsules at 5 % maltodextrin. Meanwhile, other microcapsules showed moisture contents and water activity (a_w) of 7g/100 g and 0.44 and below, respectively.

According to Sun et al. [49], the moisture content of food powders should be below 5 % to ensure prolonged shelf life and reduced microbial growth. The findings from this study were consistent as reported by Yang et al. [37], in which the moisture contents ranged between 4.15 and 5.39 g/100g. In another study, the moisture content was reported to be 5.21 % in a spray-dried pectinase-liquefied papaya powder [50]. Therefore, results in this study indicated that the low moisture contents in the microencapsulation of cellulase-treated MD-2 pineapple peel extract were affected by different carrier agents.

It was observed that water activity (a_w) exhibited significantly lowest (p < 0.05) in spray drying as compared to foam-mat drying. The microencapsulation of cellulase-treated MD-2 pineapple peel extract was affected by the type of carrier agents such as maltodextrin, which produced lower a_w in both drying conditions as compared to GA. Findings from other work involving the spray-dried pineapple pulp indicated water activities (a_w) of 0.37 [51] and 0.44 [52] using 10 % maltodextrin as the carrier agent. Similar results were shown in this study as the water activities (a_w) in spray-dried cellulase-treated MD-2 pineapple peel ranged between 0.35 and 0.44. Water activity (a_w) is the accessibility of free water in a food system that is responsible for biochemical processes [19]. Low a_w indicates a good powder quality in preventing the growth of microorganisms.

3.2. Bulk and tapped density

The bulk density in spray-drying and foam-mat drying of cellulase-treated MD2 pineapple peel powder is shown in Table 2. The bulk density for spray-dried microcapsules was highest when maltodextrin was used as a carrier agent as compared to GA. This might be due to a denser microencapsulated powder mass due to the incorporation of heavier carrier agents such as maltodextrin [49]. In addition, microencapsulation by foam-mat drying exhibited the highest bulk density (p < 0.05) as compared to spray-drying. The findings in this study were similar to those reported in microencapsulated pomegranate peel powder which the bulk density ranged between 0.30 and 0.34 g/cm³ [37]. Therefore, higher bulk density is required in industries due to reduced costs in packaging and transportation [50]. As reported by Edris et al. [53], higher bulk densities in food powders resulted in reduced storage space and oxidation prevention.

The tapped density in spray-drying and foam-mat drying for cellulase-treated MD2 pineapple peel extract powder is shown in Table 4. The tapped density for microcapsules ranged between 0.38 and 0.5 g/cm³ and 0.56–0.69 g/cm³ in spray-drying and foam-mat drying, respectively. The tapped density was observed to be influenced significantly by the carrier agent and drying techniques. Tapped density increased significantly (p < 0.05) with increasing the concentrations of maltodextrin and GA as carrier agents from 5 % to 10 % in both spray-drying and foam-mat drying. This could be explained by the impact of size enlargement, in which the size of larger particles creates spaces for smaller particles to occupy during tapping [49].

3.3. Water solubility index (WSI%)

The water solubility index (WSI) determines the solubility of powder

Table 2

Effects of different carrier agents on the physical characterization of microencapsulated MD2 pineapple peel by spray drying (150 °C) and foam-mat drying (60 °C).

Drying condition	Moisture content (g/100 g dry basis)	Water activity (a_w)	Bulk density (g/mL)	Tapped density (g/cm^3)	Water solubility index (%)	Carr Index (%)	Hausner ratio
Spray drying (150°C)							
5 % MD	4.09 ± 0.35^c	0.35 ± 0.00^d	0.39 ± 0.02^b	0.42 ± 0.02^{de}	87.30 ± 0.95^{ab}	7.50 ± 3.82^{abc}	1.08 ± 0.05^{ab}
10 % MD	6.48 ± 0.27^a	0.40 ± 0.01^{cd}	0.41 ± 0.01^b	0.50 ± 0.02^{bc}	98.53 ± 0.92^a	16.94 ± 4.13^{ab}	1.21 ± 0.06^{ab}
5 % GA	5.46 ± 0.75^{ab}	0.42 ± 0.02^c	0.32 ± 0.01^c	0.38 ± 0.01^e	75.42 ± 2.21^{bc}	15.78 ± 3.44^{ab}	1.19 ± 0.05^{ab}
10 % GA	5.89 ± 0.27^a	0.44 ± 0.00^c	0.38 ± 0.01^b	0.46 ± 0.04^{cd}	86.67 ± 11.55^{ab}	17.09 ± 6.36^a	1.21 ± 0.10^a
Foam-mat drying (60°C)							
5 % MD	5.54 ± 0.32^{ab}	0.45 ± 0.05^{bc}	0.54 ± 0.00^a	0.56 ± 0.01^b	69.40 ± 0.32^{bc}	2.62 ± 1.39^c	1.03 ± 0.01^b
10 % MD	5.58 ± 0.37^{ab}	0.42 ± 0.02^c	0.57 ± 0.04^a	0.67 ± 0.03^a	65.36 ± 8.04^c	14.99 ± 8.87^{abc}	1.18 ± 0.12^{ab}
5 % GA	4.44 ± 0.77^{bc}	0.57 ± 0.02^a	0.54 ± 0.00^a	0.56 ± 0.00^b	72.50 ± 11.7^{bc}	3.94 ± 0.61^{bc}	1.04 ± 0.01^{ab}
10 % GA	5.18 ± 0.20^{abc}	0.51 ± 0.00^{ab}	0.57 ± 0.02^a	0.69 ± 0.04^a	79.34 ± 0.59^{bc}	17.59 ± 2.13^a	1.21 ± 0.03^a

Mean with the different subscript letters in a column$^{(a-d)}$ indicates significant differences at p < 0.05. (Notes: MD: Maltodextrin, GA: Gum Arabic).

Table 3

Effects of different carrier agents on the particle size distribution of microencapsulated MD2 pineapple peel by spray drying (150 °C) and foam-mat drying (60 °C).

Drying condition	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)	Mean particle size [D4,3] (μm)	Span
Spray drying (150°C)					
5 % MD	14.84 ± 0.15^{cd}	53.71 ± 1.02^c	736.69 ± 31.2^a	259.43 ± 8.76^a	13.30 ± 0.83^a
10 % MD	11.88 ± 0.09^d	24.08 ± 0.18^d	52.89 ± 1.63^d	34.99 ± 0.81^d	1.71 ± 0.05^c
5 % GA	10.26 ± 0.32^d	19.70 ± 0.14^d	38.76 ± 0.46^d	22.41 ± 0.80^d	1.26 ± 0.05^c
10 % GA	11.24 ± 0.37^d	20.93 ± 0.33^d	40.34 ± 0.20^d	26.35 ± 4.16^d	1.38 ± 0.05^c
Foam-mat drying (60°C)					
5 % MD	20.03 ± 0.69^{bc}	99.91 ± 0.74^b	288.95 ± 32^b	164.4 ± 27.90^b	2.68 ± 0.35^b
10 % MD	21.34 ± 0.31^b	96.55 ± 0.13^b	222.00 ± 4.15^c	112.37 ± 0.40^c	2.10 ± 0.04^{bc}
5 % GA	24.90 ± 0.93^b	101.35 ± 3.71^b	237.81 ± 15.18^c	117.39 ± 1.00^c	2.10 ± 0.04^{bc}
10 % GA	31.25 ± 5.02^a	118.76 ± 5.97^a	253.28 ± 6.65^{bc}	136.40 ± 0.09^{bc}	1.87 ± 0.08^{bc}

Mean with the different subscript letters in a column$^{(a-d)}$ indicates significant differences at p < 0.05. (Notes: MD: Maltodextrin, GA: Gum Arabic).

Table 4

Bioactive volatile compounds in spray-dried microencapsulated cellulase-treated MD2 pineapple peel extract powder (10 % GA).

Peak No.	RT (min)	Compound name	Molecular formula	Percentage Peak area (%)
1	12.716	Isosorbide	C_6H10O4	21.98
2	12.716	Dianhydromannitol	C_6H10O4	21.98
3	13.694	2-Methoxy-4-vinylphenol	C_9H10O2	2.81
4	19.670	Phenol, 2,4-bis(1,1-dimethylethyl)	$C_{17}H30OSi$	45.11

RT represents retention time. Compounds were identified with more than 80 % similarity index compared to the standard mass spectra in the NIST 11 library.

to form a solution in water [15]. As shown in Table 2, WSI for all microencapsulated powders using spray drying was more than 75 % with 10 % maltodextrin at 98 %. Meanwhile, microencapsulated powders using foam-mat drying exhibited WSI within the range of 65–79 %. The effect of different carrier agents on WSI was observed to be the highest in spray drying as compared to foam-mat drying. Similar findings were observed in spray-dried rambutan peel, with a high solubility index of 98.65 % [45]. In another study, foam-mat dried pineapple powder showed WSI ranging from 55.10 to 64.05 % when maltodextrin was used as a carrier agent [39]. The result indicated that spray-dried cellulase-treated MD2 pineapple peel extract powder provides easier solubilization into various products.

3.4. Flowability and cohesiveness

The Carr index (CI) and Hausner ratio (HR) for determining the flowability and cohesiveness of microcapsules are shown in Table 2. Based on the classifications of CI (%) and HR, the flowability of <15 % CI was observed in powders at 5 % maltodextrin-spray-drying and 5 % maltodextrin-foam-mat drying, 10 % maltodextrin-foam-mat drying and 5 % GA-foam-mat drying. Meanwhile, low cohesiveness of <1.2 HR was observed in powders with treatments of spray drying using 5 % maltodextrin and GA as carrier agents. Similar results were also observed in treatments with foam-mat drying using 5 % maltodextrin, 10 % maltodextrin, and 5 % GA as carrier agents, showing low cohesiveness (<1.2) of powders. Furthermore, in a study performed by Seerangurayar et al. [54], the foam-mat freeze-dried date powder showed a fair powder flowability with CI (%) ranging between 20 and 26 %. Meanwhile, the foam-mat freeze-dried date powder using 40 % maltodextrin, 50 % maltodextrin, 40 % GA, and 50 % GA. were within the ranges of 1.25–1.35 indicating intermediate cohesiveness. Nevertheless, microencapsulated powders from pineapple peel extract carried out by Lourenco et al. [13] using spray-drying (150 °C) exhibited intermediate cohesiveness and fair flowability. Together these results indicated that the concentration of carrier agents, drying methods, and the improvement of treatments on extracts before encapsulation affect the powders' cohesiveness and flowability.

3.5. Particle size distribution

The particle size of a powder determines the products' processing, storage, and handling quality. As shown in Table 3, spray-drying of cellulase-treated MD2 pineapple peel extract powder exhibited the smallest particle size distributions [D50] and mean [D 4,3] as compared to foam-mat drying. Lower particle size resulted in a more efficient extraction of TPC. When particle size is reduced, the surface area increases making the extraction of TPC efficient. Similar results were observed in a study performed by Yang et al. [37] on the microcapsules of ciriguela peel extracts by ultrasound-assisted extraction in which spray-drying exhibited lower average diameters of microcapsules (16.75 μm) as compared to freeze-drying (25.19 μm).

These findings might be due to lower droplets of liquid feed extracts

created by the atomizer's shear, in which, low-viscosity feeds are typically used to produce spray-dried powders with lower particle sizes as opposed to high-viscosity feeds as highlighted by Ahmadian et al. [55]. Furthermore, Tupuna et al. [56] highlighted that the type or ratio/-concentrations of carrier agents do not influence the diameter size of particles. Nevertheless, the particle sizes of powders are influenced by total soluble solids (TSS%), formulation viscosity, and spray dryer operating conditions (feed flow rate, inlet temperature) [57,58]. Since spray-drying operates at a higher temperature as compared to foam-mat drying, the particle sizes of microcapsule powders are much smaller. This might be due to when treatment at higher temperatures, the heat transfer coefficient increased, leading to rapid water evaporation [53]. Thus, higher inlet temperatures allow for a reduced particle size diameter [56].

Span values in spray-dried microcapsules were lower than 2 except for 5 % maltodextrin. Which showed good solubility and homogeneity [59]. However, foam-mat drying was observed to have span values of more than 2 except for 10 % GA. According to Jiang et al. [60], span values with more than 2 resulted in the agglomerations of powder particles.

3.6. Total phenolic content

The effect of carrier agents and drying techniques on the TPC is shown in Fig. 1a and b. In this study, the type of carrier agent influenced the extract and drying techniques since GA was able to encapsulate TPC better than maltodextrin when used at 5 % (v/v). However, the control (non-cellulase treated) showed significant ($p < 0.05$) low TPC yield to extracts powder treated with cellulase (Fig. 1a and b) in both drying techniques. Similar findings observed in the microencapsulation of

ciriguela peel extract by spray-drying (150 °C) indicated higher TPC when GA was used as a carrier agent [24]. Meanwhile, a study on microencapsulation of acerola residue extract revealed higher TPC when GA was used in combination with maltodextrin as a carrier agent compared to the freeze-drying process [61]. In another study, it was observed that microencapsulation of passion fruit peel extracts by spray-drying revealed retained phenolic compounds when GA and maltodextrin were used as carrier agents.

Furthermore, high content of TPCs in GA could be found ranging from 26.79 mg GAE/g to 723.7 mg GAE/g [62]; and 2.68 mg GAE/100 g to 10.96 mg GAE/100 g [63]. According to Musa et al. [64], GA is an arabinose-galactan-protein [65] comprised of antioxidant biomolecules due to the presence of amino acid residues. Due to their unique carbohydrate-protein linked structure, GA has high efficiency as coating agent formation on phenolic compounds [66]. Findings in this study were shown to encapsulate phenolics better as the TPC was higher in spray-dried cellulase-treated pineapple peel as compared to spray-dried pineapple peel using ethanol-water extract found by Lourenço et al. [3].

3.7. Encapsulation efficiency (EE%)

The EE% of the cellulase-treated MD2 pineapple peel extract powder is presented in Fig. 2. The addition of maltodextrin and GA at both 5 % and 10 % exhibited the highest EE% of more than 89 % for all microcapsules by spray drying. Meanwhile, an effective encapsulation process due to the highest EE% (99 %) was obtained from the encapsulation of cellulase-treated MD2 pineapple peel extract by spray drying (150 °C) using 10 % GA as a carrier agent. High encapsulation efficiency could also increase product stability and prevent oxidative degradation of chemicals on particle surfaces [67]. According to Yang et al. [37], a higher EE% usually indicates less TPC on the surface of microcapsules, which could also be observed in the result. In addition, a significant increase of EE% could also be observed when the concentrations of carrier agents increased from 5 % to 10 % v/v for both maltodextrin and GA in spray drying. This may be because by increasing the concentration of carrier agents, a higher viscosity of the extracts' emulsion was produced. Thereby, when subjected to spray drying (150 °C), the MD and GA at 10 % v/v were more effective in holding the core materials (extract) inside the microcapsules [68] provided with the highest EE%. The results also showed that various studies had shown good EE% of more than 80 % in the microcapsules of enzyme-treated pistachio green hull extract [39], Georgia-grown pomegranate peel extracts [37], and maize waste extract [41] (see Fig. 3).
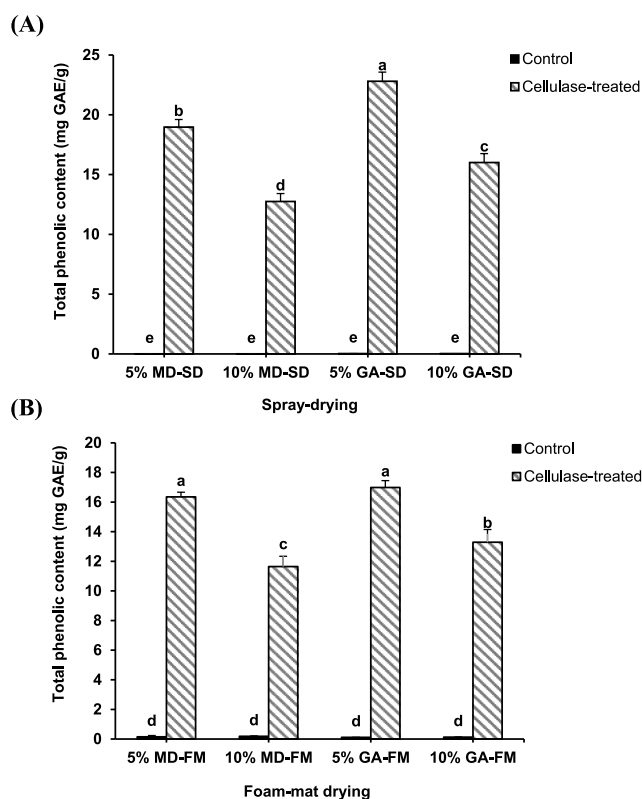
(A)

(B)

Fig. 1. Total phenolic content of cellulase-treated MD-2 pineapple peel extract powder by (A) spray drying (150 °C) and (B) foam-mat drying (60 °C) using maltodextrin and gum arabic as carrier agents. (Note; GAE: gallic acid equivalent; Control: non-cellulase-treated MD2 pineapple peel extract powder, SD: Spray-drying, FM: Foam-mat drying, MD: maltodextrin and GA: gum Arabic).
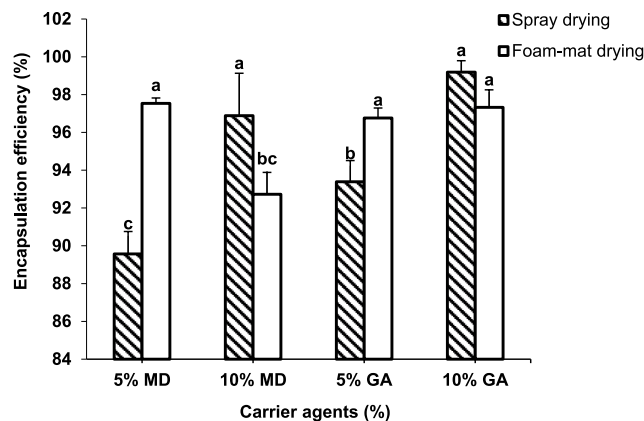

Fig. 2. Encapsulation efficiency (%) obtained from the spray-dried and foam-mat dried of cellulase-treated MD2 pineapple peel powders. (Note; GAE: gallic acid equivalent).
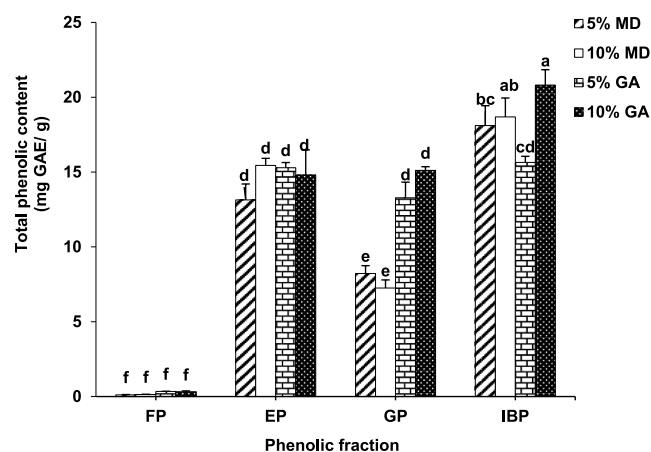
Fig. 3. Effect of different concentrations of wall materials on free, esterified, glycosylated, and insoluble-bound phenolic from spray-dried cellulase-treated MD2 pineapple peel extract powder (Notes; (Note; GAE: gallic acid equivalent, MD: Maltodextrin, GA: Gum arabic, FP: Free phenolics, EP: Esterified phenolics, GP: Glycosylated phenolics, IBP: Insoluble-bound phenolics).

3.8. Free, soluble-conjugated and insoluble-bound phenolics

Microencapsulation by spray-drying (150 °C) was chosen to further evaluate the effect of different carrier agents towards different phenolic fractions due to lower water activity (a_w), higher solubility index, higher EE%, and smaller average mean of particle size as compared to foam-mat drying. Fig. 3 shows spray drying (150 °C) on the encapsulation process of cellulase-treated MD2 pineapple peel extract. Micro-encapsulated powders using 10 % GA as carrier agent showed significantly the highest ($p < 0.05$) amount of TPC in insoluble-bound phenolics (IBP) followed by GP, EP, and finally FP. Meanwhile, the effect of different concentrations of carrier agents on spray-drying revealed 10 % GA with the highest TPC comprising all the phenolic fractions (IBP, GP, EP, and FP) amounting to 50.99 mg GAE/g. In other phenolic fractions, 5 % and 10 % GA showed a significant ($p < 0.05$) increase in the TPC from glycosylated phenolic as compared to 5 % and 10 % maltodextrin. As discussed earlier in the TPC section, GA is comprised of unique carbohydrate-protein structures that are effective in the encapsulation of phenolic compounds. These reasons might indicate that TPC was highest in IBP when using 10 % GA as a carrier agent especially as observed in glycosylated phenolics. According to one study on the encapsulation of free phenolics using extracts from sesame seeds [69], the results showed greater amounts of free phenolics than bound phenolics, demonstrating that the phenolic fraction varies depending on the agricultural waste. Furthermore, according to Elnour et al. [70], GA obtained from *acacia seygal* gum may contain sufficient amounts of non-extractable bound phenolics due to its structure being bonded to other plant materials. These reasons indicate the potential microencapsulation of cellulase-treated MD2 pineapple peel extract combined with high phenolics from GA producing bioactive-rich microcapsules. Therefore, these microcapsules can find applications as potential antioxidant agents in food systems.

Gum Arabic was chosen due to its high solubility, good biocompatibility, optimum viscosity and emulsifying agent (Kuck et al., 2016), and safety are among the most common materials that have been used as carriers for the spray-drying encapsulation of bioactive compounds [57]. The polysaccharide carrier agent is special in the capacity of encapsulating TPC has been related to its structure, being a highly branched sugar heteropolymer with small protein content, which allows protection of these compounds mainly during the critical early phase of encapsulation [61]. Based on the findings in this present study, gum Arabic showed high-quality powder characteristics compared to

maltodextrin alone during the encapsulation process. Especially in both drying methods and in the encapsulation of bound phenolics which exhibited higher when using gum arabic.

3.9. Antioxidant activities

Antioxidant activities of different phenolic fractions in cellulase-treated MD2 pineapple peel extract powder at 10 % GA are shown in Fig. 6a and b Fig. 4a and b. The microcapsule with 10 % GA was selected due to the highest total amounts of TPC in esterified phenolics (EP), glycosylated phenolics (GP), and insoluble-bound phenolics (IBP) fractions. Esterified phenolics revealed the strongest free radical scavenging ability against DPPH than GP, IBP, and FP, which were the least potent. Meanwhile, GP showed the highest significant reduction of Fe^{3+} to Fe^{2+} in the following antioxidant activities order; GP > EP > IBP > FP. Antioxidant activities presented as DPPH free radical (% inhibition) and ferric reducing antioxidant power (FRAP) exhibited stronger for other
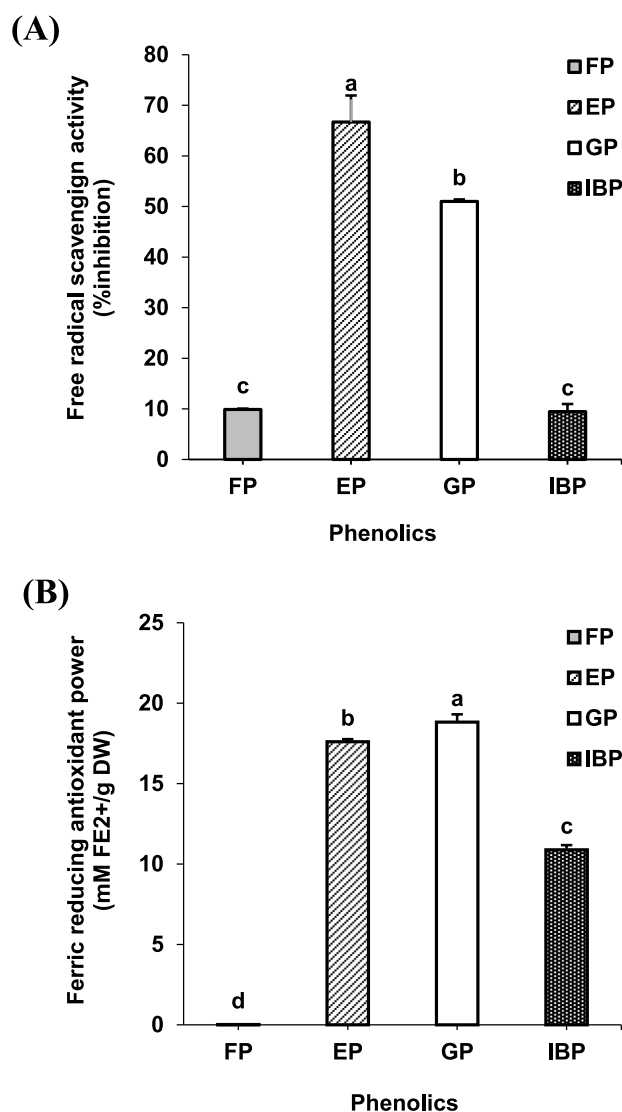
(A)

(B)

Fig. 4. Effect of spray-dried microencapsulated cellulase-treated MD2 pineapple peel extract powder (10 % GA) on the (A) DPPH free radical scavenging activity and (B) Ferric reducing antioxidant power (FRAP) of free, esterified, glycosylated and insoluble-bound phenolic (Note; FP: Free phenolics, EP: Esterified phenolics, GP: Glycosylated phenolics, IBP: Insoluble-bound phenolics).
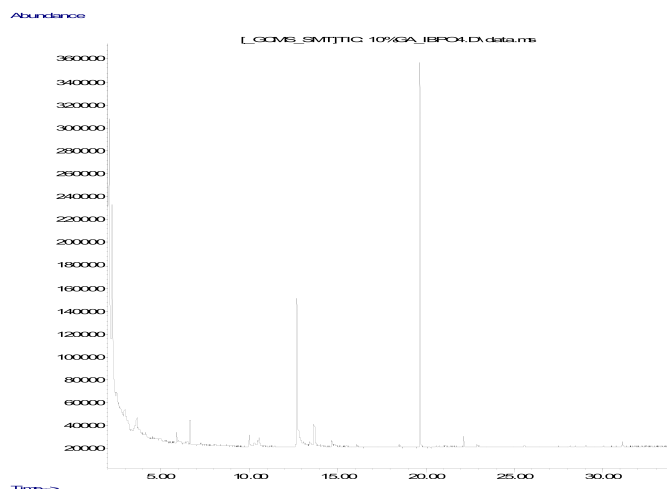
Fig. 5. Chromatograms of microencapsulated cellulase-treated MD2 pineapple peel extract powder using 10 % GA as carrier agent by spray drying at 150 °C.
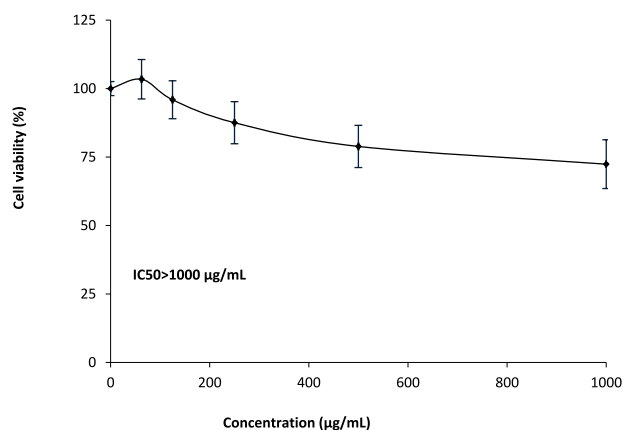

Fig. 6. Reduction of cell viability (%) of N1H3T3 fibroblast cell line against spray-dried microencapsulated cellulase-treated MD2 pineapple peel extract powder (10 % GA) for 72 h (Note; Untreated cell: control).

phenolic fractions compared to insoluble-bound phenolic which might be due to steric hindrance. Polyphenol compounds might hinder scavenging DPPH due to steric hindrance [71]. The radical scavenging of phenolic compounds might be due to the presence, position, and number of hydroxyl groups in which steric hindrance may cause a reduction of antioxidant potential in antioxidant assays [72].

Several studies highlighted the antioxidant potential of free, esterified, glycosylated, and insoluble-bound phenolics present in various agricultural by-products. Similar to the findings in this study, agricultural by-products with high antioxidants from their bound phenolics fractions were found in the fruit peels of peaches [73], blueberry and apples [74], mango [75], araticum [42], and grapes [76]. Therefore, as shown in this study, MD2 pineapple peel extract has potent antioxidant activities in microencapsulated powders due to the presence of IBP.

3.10. Bioactive volatile compounds

The GC-MS profile of bioactive volatile compounds from spray-dried cellulase-treated MD2 pineapple peel extract powder containing 10 % GA is shown in Table 4 and Fig. 5 Bioactive compounds including isosorbide (21.98 %), dianhydromannitol (21.98 %), 2-Methoxy-4-vinylphenol (2.81 %), and Phenol, 2,4-bis (1,1-dimethyl ethyl) (45.11 %)

was found in the microencapsules of cellulase-treated MD2 pineapple peel extract powder. Meanwhile, Phenol, 2,4-bis(1,1-dimethylethyl) was discovered to be the predominant compound by more than 45 %. Based on the previous finding on the identification of bioactive volatile compounds in cellulase-treated MD-2 pineapple peel extract, Phenol, 2,4-bis(1,1-dimethylethyl) presented in the phenolic fraction extracts were free (13.68 %), esterified (14.84 %), glycosylated (89.80 %) and IBP (0.51 %) [35]. The findings from this study revealed how Phenol, 2, 4-bis (1,1-dimethyl ethyl) achieved the largest percentage peak area (45.11 %) in microcapsule powder due to the presence of all phenolic fractions indicated in findings of a similar treatment on cellulase-treated MD2 pineapple peel extracts [35]. Meanwhile, only the esterified phenolic fraction (0.16 %) contributed to the existence of 2-Methoxy-4--vinylphenol from earlier findings in a comparable enzyme treatment of extracts [35], which might explain why the strong antioxidant from EP demonstrated in this study.

In another study, GC-MS profiling of *L. aequata* leaves revealed the presence of phenol, 2,4-bis(1,1-dimethylethyl), which has high antioxidant and anticancer action [77,78]. Mostofa et al. [77] reported that by molecular docking of Phenol, 2,4-bis (1,1-dimethylethyl) compound against urate oxidase (PDB: 1R4U) and glutathione reductase (PDB: 3GRS), studies revealed anticancer activity as a possible pharmaceutical agent for the treatment of many disorders. Meanwhile, avocado roots exhibited the production of Phenol, 2,4-bis (1,1-dimethylethyl) when induced with salicylic acid which promotes resistance against *Aspergillus* and *P. cinnamomi* [79]. Thereby, indicating the compound as antifungal [80].

Another important compound to note is 2-Methoxy-4-vinylphenol which provides significant importance as one of the aroma compounds in Xinjiang dried figs [43]. In another study, red cabbage extract was found to contain the compound 2-Methoxy-4-vinylphenol which has potent antioxidant and antimicrobial activity [81]. The compound 2-Methoxy-4-vinylphenol was found to inhibit hyper-phosphorylation of Rb (tumor suppressor protein) induced by Benzo[a]pyrene (BaP) as reported by Jeong & Jeong [82]. Therefore, findings from microcapsule powders with the presence of bioactive-rich extracts might find applications as antioxidant, antifungal, antiinflammatory, and anticancer agents.

3.11. Cytotoxicity activity

The cytotoxicity activity of cellulase-treated MD2 pineapple peel extract powder at 10 % GA is shown in Fig. 6. The IC_{50} values were more than 1000 μg/mL which indicated low in toxicity effect of extract powder against NIH3T3 fibroblast cells. Similar findings on encapsulated pomegranate peel extract showed no cytotoxicity effect on NIH3T3 fibroblast cells in applied concentration [83]. In this study, insoluble-bound phenolics in 10 % GA pineapple peel extract powder play a significant role as a bioactive compound demonstrating no cytotoxicity activity present. This could be supported by various research where bound phenolics demonstrated potential anti-obesity, antidiabetic, and anticancer properties exhibited by pomegranate seeds [84], and Sophia seed meals [85]. Meanwhile, the compound phenol, 2,4-bis(1,1-dimethylethyl) present in the powder indicates a potential anticancer agent as highlighted by Mostafa et al. [77] in which the compound was also present in *Leea aequata* L. leaves. The microencapsulated powder extract (10 % GA) has potential use in food products such as antioxidant as well as functional ingredients in meat and fish, dough, bakery, and confectionaries. Meanwhile, the presence of a 2-Methoxy-4-vinylphenol compound indicates the potential of the powder microcapsule as a flavoring agent. Based on these findings, microencapsulated MD2 pineapple peel extract has the potential as an anticancer, anti-obesity, and antidiabetic agents which have applications as neutraceuticals, functional foods, pharmaceuticals, and cosmetics ingredients.

3.12. Conclusions

Microencapsulation by both spray-drying and foam-mat drying revealed high-quality powder characteristics as observed from pineapple peel extract powders. Spray-dried samples exhibited lower water activity, higher solubility, lower particle size, and span values less than 2 indicating homogenized powders. The most effective carrier agent in the encapsulation of phenolic compounds was observed using 10 % GA, especially in the encapsulation of insoluble-bound phenolics. Antioxidant activities revealed that EP and GP were the highest in scavenging DPPH free radicals and reducing FRAP followed by both IBP and FP. Bioactive volatile compounds identified in 10 % GA comprised of 2-Methoxy-4-vinylphenol and Phenol, 2,4-bis(1,1-dimethylethyl) which exhibited inactive cytotoxicity activity towards the NIH3T3 fibroblast cell line. The compound Phenol, 2,4-bis(1,1-dimethylethyl) represented the overall phenolic fractions (FP, EP, GP, and IBP) identified from the GCMS profile. The identification of bioactive compounds in the microencapsulated extract powder showed potential as an ingredient in functional foods, pharmaceuticals, and cosmetics. Future exploration of MD2 cellulase-treated MD2 pineapple peel extract powder should be further analyzed on its anticancer, and antidiabetic properties, bioavailability, bioaccessibility in the human gastrointestinal digestive system, the molecular docking to identify the mechanisms of the compounds' action as drugs for various diseases.

CRediT authorship contribution statement

Nur Liyana Nordin: Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Jamilah Bakar:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Noranizan Mohd Adzahan:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Ahmad Faizal Abdull Razis:** Investigation. **Norsharina Ismail:** Investigation. **Rabiha Sulaiman:** Writing – review & editing, Supervision, Methodology, Conceptualization.

Ethical approval

Not applicable.

Code availability

Not applicable.

Funding

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Declaration of competing interest

Authors declared there is no conflict of interest.

Data availability

The data that has been used is confidential.

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