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Preparation and Physical Evaluation of Maltodextrin-Assisted Rice Bran Extract Granules with Targeted Qualitative LC-MS/MS Screening

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Rice bran is a potential source of phenolic and flavonoid compounds for the development of value-added formulations. This exploratory study examined two ethanol-extraction procedures and evaluated maltodextrin-assisted freeze-dried formulations for powder recovery, residual moisture content, physical appearance and qualitative water dispersibility before wet granulation. Under the conditions tested, maceration yielded 22.50% crude extract, compared with 7.00% for Soxhlet extraction. These yields were treated as descriptive findings, since solvent concentration, extraction temperature and extraction duration differed between the two procedures. Among the maceration-based formulations, the crude extract:maltodextrin ratio of 10:30:100 gave the highest powder recovery (29.5%), while the 10:20:100 formulation had the lowest residual moisture content (3.18%), a fine, dry powder appearance and good qualitative water dispersibility. Qualitative LC-MS/MS screening of a separately analyzed pre-formulation extract (laboratory sample code 1A) yielded laboratory-reported assignments for ferulic acid, 3,4-dihydroxybenzoic acid, gallic acid, quercetin and luteolin-7-O-glucoside. The analytical method was not quantitatively validated and the corresponding formulations were not analyzed after freeze-drying or granulation. The retention of these compounds during processing, biological activity and storage stability remain unverified. These preliminary findings provide a basis for further formulation studies involving independent experimental replicates, validated quantitative analysis and systematic assessment of physico-chemical stability and functional properties.

Keywords: Rice bran, Maltodextrin, Freeze drying, Formulation screening, Granulation, LC-MS/MS, Phenolic compounds.

INTRODUCTION

Rice (*Oryza sativa* L.) is one of the most important cereal crops worldwide and serves as a staple food for more than half of the global population, particularly in Asian countries. During rice processing, substantial quantities of rice bran are generated as byproducts. Although often considered a low-value agricultural residue, rice bran is recognized as a rich source of bioactive phytochemicals including phenolic acids, flavonoids, γ-oryzanol, tocopherols and tocotrienols [1-6]. These compounds have attracted considerable attention due to their antioxidant, anti-inflammatory, cardioprotective and neuroprotective properties [1-4,7-9]. Consequently, rice bran has emerged as a promising raw material for developing functional foods, nutraceuticals and health-promoting products.

Among the phytochemicals present in rice bran, phenolic acids and flavonoids are considered major contributors to its biological activities. Previous studies [10-18] have reported that the rice bran contains various phenolic acids and flavonoids including 4-hydroxybenzoic acid, 3,4-dihydroxybenzoic acid, caffeic acid, ferulic acid, syringic acid, apigenin, vitexin, isorhamnetin and naringenin. These compounds exhibit strong free-radical-scavenging activity and may help prevent oxidative stress-related disorders [10-13,17-19]. The recovery and utilization of such bioactive constituents from rice bran, therefore, represent an attractive strategy for increasing the value of rice-processing by-products while supporting sustainable resource utilization. Despite its phytochemical richness, concentrated rice bran extract can be difficult to handle due to its hygroscopicity, stickiness and poor powder formation [20-

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24]. The carrier assisted drying and granulation may improve physical handling, powder recovery and dispersibility.

Freeze drying is widely recognized as an effective dehydration technique for preserving thermolabile or heat-sensitive phytochemicals since water is removed by sublimation under low-temperature and reduced-pressure conditions [20-26]. Compared with conventional drying methods, freeze drying minimizes thermal degradation and helps maintain the chemical integrity of phenolic and flavonoid compounds. Furthermore, the addition of carrier agents such as maltodextrin can improve powder recovery, reduce stickiness, enhance solubility or dispersibility and protect phenolic compounds during drying and storage [20-27]. Freeze-dried powders can subsequently be transformed into granules with improved flowability, handling characteristics and formulation performance, making them suitable for industrial-scale applications.

LC-MS/MS is widely used to screen phenolic acids and flavonoids in rice, rice bran and other complex plant matrices due of its sensitivity and selectivity [14-18,28-30]. Multiple reaction monitoring can support targeted detection of selected compounds, while reliable identification depends on the availability of appropriate reference standards and supporting mass spectrometric evidence. This study examined two ethanol-extraction workflows for rice bran and evaluated maltodextrin-assisted freeze-dried extract formulations based on powder recovery, residual moisture content, physical appearance and qualitative water dispersibility prior to wet granulation. The qualitative LC–MS/MS screening of a separately recorded pre-formulation extract was used to report preliminary assignments of selected phenolic and flavonoid compounds.

EXPERIMENTAL

Rice bran was obtained from Thamna Praneet Kkao Insee Sri Samran, Khon Sawan District, Chaiyaphum Province, Thailand. Analytical-grade maltodextrin, ethanol, methanol and formic acid were used throughout the study. Sweeteners, flavouring agents and a binder were employed in granule formulation. Ultrapure water was used for all experiments.

Preparation of rice bran extracts: Rice bran was passed through a 500 μm sieve to obtain a uniform particle size prior to extraction. The moisture content of the sieved rice bran was 12.47%. Rice bran extracts were prepared using maceration and Soxhlet methods and the crude-extract yields obtained under the respective applied conditions were recorded descriptively. For the maceration method, rice bran was extracted with 80% (v/v) ethanol at room temperature for 72 h, with mechanical stirring for 30 min once daily. The extract was filtered through muslin cloth and concentrated under reduced pressure using a rotary evaporator at 40 ºC. For Soxhlet extraction, rice bran was extracted with 95% ethanol for 8 h using a Soxhlet apparatus. The resulting extract was filtered and concentrated under reduced pressure at 40 ºC. Ethanol and aqueous ethanol were selected since hydroalcoholic solvents are commonly used for extracting the phenolic and flavonoid constituents from rice bran and other plant matrices [10-13,19]. The extraction yield was calculated according to the following equation:

$$\text{Yield (\%)} = \frac{\text{Weight of dried extract}}{\text{Weight of dry rice bran}} \times 100$$

The two extraction workflows differed in ethanol concentration, extraction temperature and extraction duration. They were therefore treated as distinct process conditions rather than as a controlled comparison of extraction method. The measured yields were used descriptively to guide material selection for subsequent formulation work.

Screening of maltodextrin-assisted freeze-dried formulations: The concentrated extracts were mixed with maltodextrin and distilled water at the ratios shown in Table-1. All formulations were frozen at -20 ºC for 48 h and freeze-dried for ~24 h using the same programmed cycle, comprising primary drying from -35 to 0 ºC followed by secondary drying from 5 to 30 ºC under vacuum. The recovered materials were weighed and evaluated for moisture content, physical appearance and qualitative water dispersibility. This experiment screened formulation ratios under a fixed drying cycle; freezing rate, shelf temperature profile, chamber pressure and drying duration were not independently optimized.

Preparation of rice bran granules: Freeze-dried rice bran extract powder obtained under the selected freeze-drying condition was used as the primary source of phytochemicals for granule formulation. The powder was blended with sweetener, flavouring agents and binder until a homogeneous mixture was achieved. Subsequently, 99.8% ethanol (80-100 mL) was gradually added under continuous mixing to obtain a cohesive wet mass. The resulting wet mass was passed through a No. 20 sieve to obtain uniform wet granules, then dried in a hot-air oven at 45-60 ºC for 1-2 h. After drying, the granules were passed through a No. 16 sieve to obtain particles smaller than 1.20 mm. The prepared granules were stored in airtight containers at room temperature for physical observation. No LC-

TABLE-1
FORMULATION SCREENING OF RICE BRAN EXTRACTS USING DIFFERENT EXTRACT–MALTODEXTRIN–WATER RATIOS

Sample extraction	Extraction method	Crude:MD: Water (g)	Weight before freeze drying (g)	Weight after freeze drying (g)	Powder recovery (%)	Physical appearance	Moisture (%)
Rice bran	Soxhlet	10:0:10	11.04	0.75	6.8	Yellow powder	5.82
		10:10:10	12.03	1.54	12.8	Fine powder	4.15
		10:20:10	13.00	2.67	20.5	Fine powder	2.93
		10:30:10	14.09	3.52	25.0	Thick cream	14.21
	Maceration	10:0:100	110.02	7.48	6.8	Started to turn into powder	6.75
		10:10:100	120.06	23.69	19.7	Dry powder	4.42
		10:20:100	130.10	27.35	21.0	Dry powder	3.18
		10:30:100	140.02	41.32	29.5	Yellow powder	5.82

MS/MS measurement, biological-function assay or storage-stability study of the final granules was performed.

Qualitative LC-MS/MS screening: The qualitative LC-MS/MS screening was performed on a separately recorded pre-formulation rice bran extract designated as laboratory sample code 1A using an AB SCIEX 3200 QTRAP operated in negative electrospray ionization multiple-reaction monitoring mode. According to the laboratory record, extract 1A was prepared using 80% ethanol for 24 h, filtered through a nylon membrane with a reported pore size of 0.25 μm and diluted two-fold with methanol. Chromatographic separation was performed using a CORTECS C_{18} column (100 mm × 2.7 mm, 2.6 μm) under isocratic elution with 0.1% formic acid in water:methanol (20:80, v/v) at a flow rate of 0.3 mL/min and a column temperature of 20 ºC. The shared procedure specified an injection-volume range of 15-20 μL. The source settings were as follows: curtain gas, 25 psi; nebulizer gas, 50 psi; heating gas, 50 psi; source temperature, 550 ºC; and ion-spray voltage of 4500 V. This analytical record was distinct from the 72 h maceration workflow used in the formulation experiment. Accordingly, the LC-MS/MS results were interpreted solely as qualitative screening data for sample 1A and not as evidence of phytochemical retention in the formulated granules.

The predefined screening panel and available ion-pair information are provided in Table-2. Laboratory-reported assignments were based on agreement between the sample and reference-standard retention times within ± 0.50 min, together with precursor-ion and diagnostic product-ion evidence.

Data analysis: Extraction and formulation outcomes were analysed descriptively using recorded process weights, calculated extraction yields and powder recoveries, measured moisture content and qualitative observations of physical appearance and water dispersibility. Independent replicate batches were not available to estimate variability or conduct inferential statistical analysis. Accordingly, no *p*-values or claims of statistical significance are reported.

TABLE-2
REFERENCE ION-PAIR PARAMETERS FOR THE PHYTOCHEMICAL SCREENING PANEL

Analyte in reference panel	Precursor *m/z*	Product ion *m/z*
4-Hydroxybenzoic acid	136.879	92.912
3,4-Dihydroxybenzoic acid	152.875	107.856
Cinnamic acid	146.938	102.942
Ellagic acid	300.886	145.067
Gallic acid	168.881	124.954
Gentisic acid	152.891	107.862
trans-4-Hydroxycinnamic acid	162.932	118.937
Caffeic acid	178.910	133.694
trans-Ferulic acid	192.910	133.969
Luteolin	284.911	131.820
Luteolin 7-glucoside	447.094	284.927
Catechin	288.965	244.928
Epicatechin	288.957	123.000
Apigenin	268.906	150.909
Quercetin	300.919	179.000
Hesperidin	609.219	300.940
Vitexin	431.000	311.186
Myricetin	316.914	136.933
Rutin	609.235	270.988
Naringenin	270.936	150.890
Kaempferol	284.876	158.945
Isorhamnetin	314.999	299.910
Emodin	268.912	225.119

RESULTS AND DISCUSSION

The extraction yields obtained from rice bran using maceration and Soxhlet extraction methods are given in Table-3. Under the specific conditions tested, the maceration method produced 225 g of crude extract from 1,000 g of rice bran (22.50%), whereas the Soxhlet workflow produced 5.6 g from 80 g (7.00%). The extraction methods differed in ethanol concentration (80% *versus* 95%), temperature and extraction duration (72 h at room temperature *versus* 8 h under Soxhlet heating). Consequently, the observed difference in crude extract yield cannot be attributed to the extraction method alone as solvent composition and temperature may have influenced the extraction efficiency. Aqueous ethanol can facilitate the extraction of polar and moderately polar constituents from plant materials [10-13,19]. Rice bran contains phenolic acids, flavonoids, γ-oryzanol derivatives and other phytochemicals with varying polarities. The higher crude extract yield obtained by maceration method may reflect the combined effects of solvent composition and extraction conditions. Prolonged exposure to elevated temperatures during Soxhlet extraction may affect the stability of certain heat-sensitive constituents [20,23,24,26]. Thus, the maceration extract was selected for subsequent formulation studies since it provided a higher quantity of crude extract under the conditions tested.

Screening of maltodextrin-assisted freeze-dried formulations: The effects of maltodextrin concentration on the freeze-drying performance of rice bran extracts are presented in Table-1. Freeze drying was used to convert the concentrated extract into a powder for subsequent formulation studies. Maltodextrin was added as a carrier to facilitate powder formation and potentially reduce stickiness during drying [20-24,27].

Under the same drying cycle, powder recovery increased from 6.8% without maltodextrin to 19.7%, 21.0% and 29.5% at crude extract:maltodextrin:water ratios of 10:10:100, 10:20:100 and 10:30:100, respectively. These observations indicate a progressive increase in recorded powder recovery with increasing maltodextrin concentration under the tested condi-

TABLE-3
CRUDE EXTRACT YIELD OF RICE BRAN OBTAINED BY MACERATION AND SOXHLET EXTRACTION

Sample extraction	Sample quantity (g)	Amount of solvent (mL)	Weight of extract after evaporation (g)	Yield of the extract after evaporation (%)
Rice bran (Maceration)	1,000	5,000	225	22.50
Rice bran (Soxhlet)	80	400	5.6	7.00

tions. The increase in recovery may reflect the contribution of maltodextrin to powder formation and handling, although these effects were not independently evaluated.

Moisture content varied across the formulations. The 10:20:100 formulation exhibited the lowest measured moisture content (3.18%), whereas the 10:30:100 formulation had a higher value of 5.82%. The 10:20:100 formulation was selected for granulation based on its low measured moisture content, dry and fine-powder appearance and good qualitative water dispersibility. Although the 10:30:100 formulation provided the highest powder recovery, the 10:20:100 formulation was preferred based on the combined physical characteristics observed during screening.

Physical characteristics and water dispersibility: The physical appearance and water dispersibility of freeze-dried rice bran extracts prepared at different crude extract:maltodextrin:water ratios are shown in Tables 4 and 5. The visual assessment revealed differences in powder formation and dispersion behaviour among the tested formulations.

For the Soxhlet extract, the formulation without maltodextrin (10:0:10) showed poor powder formation and remained sticky on the drying tray. It formed visible aggregates upon dispersion in water. The addition of maltodextrin improved powder formation and dispersion, with the 10:10:10 and 10:20:10 formulations producing fine powders that dispersed more uniformly. The 10:30:10 formulation formed a cream-like product rather than a free-flowing powder, which indicate an inadequate powder formation under the applied drying conditions.

For the maceration extract, the formulation without maltodextrin (10:0:100) formed only a partial powder and showed poor dispersion, with visible undissolved particles. The addition of maltodextrin improved the physical appearance and dispersion behaviour. The 10:10:100 and 10:20:100 formulations formed more homogeneous suspensions with fewer visible aggregates. The 10:30:100 formulation produced a uniform yellow powder with good visual dispersibility, although its measured moisture content was higher than that of the 10:20:100 formulation. These observations are consistent with the reported use of maltodextrin as a carrier to facilitate powder formation and improve the handling of plant extracts [20-24,27]. Among the maceration formulations, the 10:20:100 ratio was selected for subsequent granule preparation based on its dry powder appearance, lower measured moisture content and satisfactory water dispersibility. This selection reflects the qualitative assessment of the tested formulations rather than a validated optimum.

Qualitative LC-MS/MS screening of separately recorded rice bran extract 1A: Selected MRM chromatograms of rice bran extract 1A before formulation are presented in Fig. 1. The qualitative LC-MS/MS screening tentatively assigned five compounds *viz*. 3,4-dihydroxybenzoic acid (1.20 min), quercetin (1.20 min), ferulic acid (1.13 min), gallic acid (1.13 min) and luteolin-7-O-glucoside (1.07 min). These assignments are based on the recorded chromatographic data and the corresponding ion transitions are listed in Table-6.

Conclusion

Rice bran extract was successfully processed into maltodextrin-assisted freeze-dried powders and granules at a small scale. The maceration and Soxhlet extraction methods yielded 22.50% and 7.00%, respectively, although differences in the solvent concentration, temperature and extraction duration prevent direct attribution of the yield difference to the extraction method alone. Among the maceration formulations, the 10:30:100 ratio provided the highest recorded powder recovery (29.5%), whereas the 10:20:100 ratio exhibited the lowest measured moisture content (3.18%), a fine-powder appearance and good qualitative water dispersibility. The latter was selected for granulation based on these characteristics. The qualitative LC-MS/MS screening of a separately recorded extract (sample 1A) yielded tentative assignments for five phytochemicals. The findings are preliminary, as independent batch replication, quantitative analytical validation, matched pre- and post-processing measurements and stability studies were not available.

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TABLE-4
PHYSICAL PROPERTIES AND WATER DISPERSIBILITY OF RICE BRAN POWDERS PREPARED USING DIFFERENT CRUDE EXTRACT–MALTODEXTRIN–WATER RATIOS UNDER IDENTICAL FREEZE-DRYING CONDITIONS

Samples	Extraction	Crude:MD:Water (g)	Physical characteristics	Water dispersibility*
Rice bran	Soxhlet	10:0:10	Sticky mass	Poor
		10:10:10	Soft powder	Moderate
		10:20:10	Fine powder	Good
		10:30:10	Fine powder	Good
	Maceration	10:0:100	Coarse powder	Moderate
		10:10:100	Sticky mass	Poor
		10:20:100	Dry fine powder	Good
		10:30:100	Uniform yellow powder	Good

*Water dispersibility was classified as poor, moderate, or good based on the degree of powder dispersion and the presence of visible aggregates after mixing with distilled water.

TABLE-5
PHYSICAL CHARACTERISTICS AND QUALITATIVE WATER DISPERSIBILITY OF RICE BRAN POWDERS PREPARED USING DIFFERENT CRUDE EXTRACT:MALTODEXTRIN:WATER RATIOS UNDER A FIXED FREEZE-DRYING CYCLE

Samples	Extraction	Crude:MD:Water (g)	Physical characteristics and water dispersibility	Caption note
Rice bran	Soxhlet	10:0:10	–	Sticky, non-dried mass poorly formed large aggregates and dispersed incompletely in water
		10:10:10		Soft yellow powder, moderately dispersed with visible aggregates
		10:20:10		Fine powder, well-dispersed relatively uniformly with minor sediment
		10:30:10		Fine powder, well- readily dispersed in water with minimal aggregation
	Maceration	10:0:100		Coarse powder, moderately dispersed in water, but produced a turbid suspension with visible particles
		10:10:100		Sticky mass, poorly formed, a viscous suspension, and dispersed incompletely
		10:20:100		Dry fine powder, well-dispersed readily in water with minimal aggregation
		10:30:100		Uniform yellow powder, well-dispersed in water with minimal visible particles

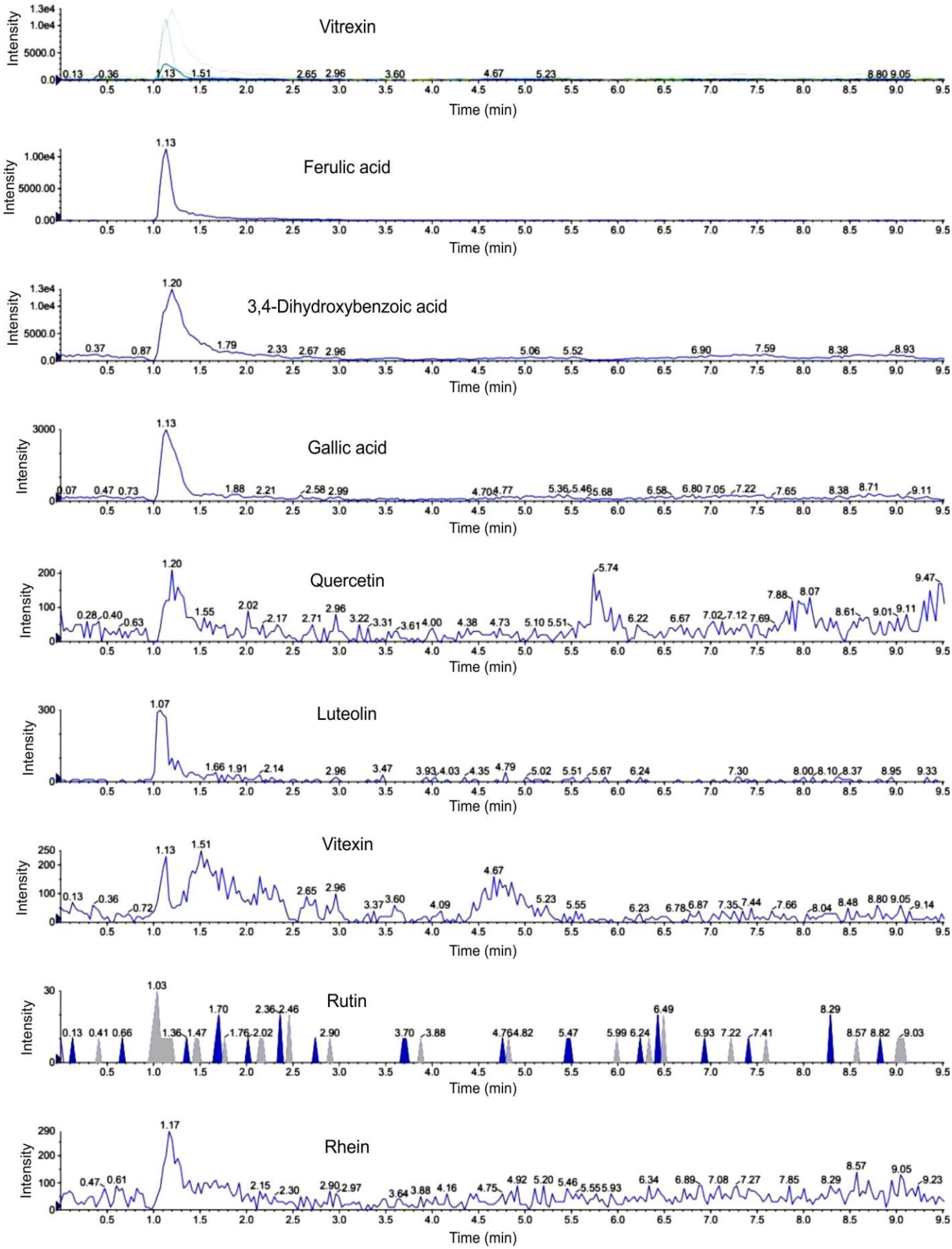


Fig. 1. Selected MRM chromatograms of rice bran extract 1A

TABLE-6
LC–MS/MS SCREENING DATA OF PHENOLIC COMPOUNDS IN RICE BRAN EXTRACT 1A

Compound assignment	Displayed MRM (*m/z*)	Sample RT (min)	Laboratory outcome
Ferulic acid	192.910 → 133.969	1.13	detected
3,4-Dihydroxybenzoic acid	152.889 → 108.929	1.20	detected
Gallic acid	168.881 → 124.954	1.13	detected
Quercetin	300.919 → 150.800	1.20	detected
Luteolin-7-O-glucoside	447.094 → 284.927	1.07	detected
Vitexin	431.000 → 310.800	Not assigned	Not detected
Rutin	609.246 → 299.973	Not assigned	Not detected
Rhein	282.892 → 238.824	Not assigned	Not detected

CONFLICT OF INTEREST

The authors declare that there is no conflicts of interest regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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