Development of Flowable Granules from *Terminalia bellirica* Waste for Health Product Applications

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Abstract

Terminalia bellirica (Gaertn.) Roxb., a member of the Combretaceae family. The ripe fruit of this plant contains key active tannins such as chebulagic acid, ellagic acid, and gallic acid, contributing to its antioxidant, anti-inflammatory, antibacterial, hepatoprotective, and antidiabetic properties. Despite its extensive use in health products, the extraction process generates substantial waste from the pulp and seeds, which still retain beneficial properties. This study aims to valorize this waste by developing a formulation and process to convert the sticky byproduct into a flowable material suitable for health product manufacturing. The study evaluated the effects of different diluents on the physical properties of powdered *T. bellirica* extract. The fruit was extracted through boiling and filtration, followed by drying and granulation using diluents such as maltodextrin, microcrystalline cellulose, and lactose monohydrate. The resulting granules were assessed for appearance, moisture content, angle of repose, bulk density, and tapped density. Results showed that all three diluents successfully produced granules with excellent flow properties. The formulations exhibited fine, non-caking brown powders with moisture content between 6–8%, indicating good to excellent flowability. These findings suggested that the developed granules possess ideal characteristics for use as raw materials in the production of herbal and health-related products, offering a sustainable approach to waste utilization in the extraction industry.

Keywords: *Terminalia bellirica, maltodextrin, microcrystalline cellulose, lactose monohydrate, granulation technique*

1. Introduction

The extraction industry generates significant amounts of byproducts, often leading to waste disposal challenges and environmental concerns. *Terminalia bellirica* (Gaertn.) Roxb. (SAMO PHI-PHEK), a medicinal plant from the Combretaceae family, is widely utilized for its bioactive compounds, including chebulagic acid, ellagic acid, and gallic acid (Sobeh et al., 2019), which exhibit antioxidant (Gupta et al., 2021), anti-inflammatory (Gupta et al., 2021), antibacterial (Dharmaratne et al., 2018), hepatoprotective (Gupta et al., 2021), and antidiabetic properties (Gupta et al., 2020). Its ripe fruit is used to reduce fever and has a mild laxative effect, helping to relieve constipation (Kumar, & Khurana, 2018). Despite its therapeutic potential, the extraction process results in large quantities of discarded pulp and seeds, which still contain valuable bioactive compounds. Diluents are essential in the preparation of dry powders and granules, ensuring optimal moisture content and flow properties. Commonly used diluents include microcrystalline cellulose (Kwari et al., 2023), lactose monohydrate (Son et al., 2019), and maltodextrin (Leticia, & Gómez Martín, 2019), which enhance the physical characteristics and processing efficiency of granules. However, rather than being efficiently utilized, these byproducts are often treated as waste due to their sticky nature and lack of processability, limiting their direct application in health product manufacturing. However, if properly processed, this waste could serve as a valuable raw material for developing herbal and functional health products. Therefore, this study aims to convert *T. bellirica* extraction waste into a flowable material suitable for production processes. By optimizing the use of diluents in the formulation, the study seeks to enhance the usability of these byproducts while promoting sustainability and resource efficiency in the extraction industry.

The findings from this research will contribute to reducing industrial waste, maximizing the benefits of *T. bellirica*, and supporting the development of innovative health products.

2. Objectives

The objective of this study is to evaluate the effects of the type and concentration of diluents on the physical properties of powdered *T. bellirica* extract.

3. Materials and Methods

The *T. bellirica* (ripe fruit) was obtained from Sun Herb Thai Chinese Manufacturing Plant, College of Pharmacy, Rangsit University, Pathum Thani, Thailand. The raw materials including maltodextrin, microcrystalline cellulose, and lactose monohydrate were purchased from Krungthep Chemi Company Limited, Bangkok Thailand.

Extraction and Powdered Preparation Procedure

A total of 130 g of *T. bellirica* fruit was heated with 800 mL of water at 85±5°C for 4 hours to simulate an industrial extraction process. The mixture was then filtered to separate the supernatant from the biomass. The collected biomass was subsequently boiled at 85±5°C until the pulp softened and the seeds were completely removed.

The biomass was further processed into powdered extracts using three excipients at varying concentrations: (1) maltodextrin, (2) microcrystalline cellulose, and (3) lactose monohydrate. The powder preparation process began with mixing the biomass with the selected excipient at the desired concentration to form a damp mass. Wet granulation was performed by passing the mixture through a mesh No. 20 sieve. The granules were then dried in a hot-air oven at 50±5°C for 4 hours. After drying, the granules were ground and sieved through a mesh no. 60 to obtain a fine powder. The final yield percentage of the nine granule formulations was subsequently determined (Table 1).

Table 1 Yield of the *T. bellirica* powdered extraction.

Formulation	Sample	Granulation yield (% w/w)
T1	10.0% Maltodextrin	35.77
T2	15.0% Maltodextrin	48.80
T3	20.0% Maltodextrin	53.50
T4	10.0% Microcrystalline	48.46
T5	15.0% Microcrystalline	53.72
T6	20.0% Microcrystalline	57.23
T7	10.0% Lactose Monohydrate	50.59
T8	15.0% Lactose Monohydrate	58.76
T9	20.0% Lactose Monohydrate	65.08

Physical properties evaluation

Appearance

Visual inspection was performed using organoleptic techniques to assess parameters such as color, shape, fineness, odor, and dryness.

Moisture content

The moisture content of all nine granule formulations was determined using a Moisture Balance (Radwag MAC50/NH, Poland). For each measurement, 1 g. of the sample was placed on the instrument tray and heated at a temperature not exceeding 120°C for up to 10 minutes. The moisture balance automatically displayed the moisture content as a percentage. Each formulation was analyzed in triplicate to ensure accuracy and reproducibility.

Angle of Repose

The flowability of the granules was assessed by measuring the angle of repose. The powder was poured through a glass funnel positioned 5 cm above the surface forming a conical pile. The height (h) and radius (r) of the cone were recorded, and the angle of repose (θ) was calculated using Equation (1):

$$\text{Angle of Repose } (\theta) = \tan^{-1}(h/r) \quad (1)$$

where Angle of Repose (θ), h and r = height (cm) and radius (cm) of the powder cone.

Test for Bulk and Tapped Density

To determine the bulk density (Saingam et al., 2015), 10 mL of granule powder was carefully placed into a 50 mL graduated cylinder, and the initial volume was recorded. The cylinder was then tapped gently on a padded surface such as a cloth and book to prevent damage. Tapping was repeated 100 times, and the volume was recorded. If the volume continued to decrease, the tapping procedure was repeated for another 100 taps, and the new volume was recorded. This process was continued until the volume no longer decreased, at which point the final volume was recorded. Each formulation was tested in triplicate to ensure accuracy. The bulk density was then calculated using Equation (2):

$$\text{Bulk density} = m/V_0 \quad (2)$$

where m = mass (g), V_0 = Bulk volume (ml)

Tapped density (Saingam et al., 2015) was evaluated to assess the packing capacity and density of the powder. After determining the bulk density, the same 50 mL graduated cylinder filled with 10 mL of granule powder was tapped 100 times at a height of 5-6 cm, with the same tester performing the procedure consistently for all samples to ensure reliability and accuracy, as described in the bulk density procedure. The final volume was recorded once the volume no longer decreased with further tapping.

Each formulation was tested in triplicate. Tapped density was then calculated using Equation (3):

$$\text{Tapped density} = m/V_f \quad (3)$$

where m = mass (g), V_f = Tapped volume (ml)

To assess the powder's cohesiveness and flowability trend, the Compressibility Index was calculated using Equation (4) (Saingam et al, 2015) as follows:

$$\text{Compressibility index} = ((V_0-V_f)/V_0) \times 100 \quad (4)$$

The Hausner ratio was then calculated using Equation (5) (Saingam et al., 2015) to evaluate the flowability of the granules. The Hausner ratio is given by:

$$\text{Hausne Ratio} = V_0/V_f \quad (5)$$

A higher Hausner ratio indicates poorer flowability, while a lower value suggests better flow characteristics. Both the compressibility Index and Hausner ratio are critical for evaluating the packing behavior, cohesiveness, and flow properties of the granules.

Figure 1 Physical characteristics of the 9 powdered extract formulations.

T1 (a), T2 (b), T3 (c), T4 (d), T5 (e), T6 (f), T7 (g), T8 (h) and T9 (i) (photographed by camera).

4. Results and Discussion

The physical characteristics of the powdered extract varied from light to dark brown, with a fine powder texture, a characteristic odor, and a dried granule form (Figure 1). The percentage yield of the dry extract powder preparation is summarized in Table 1, which shows that an increase in the proportion of diluent correlates with a higher yield percentage. It was noted that the use of lactose monohydrate as a diluent resulted in a higher yield compared to the other two diluents at the same concentration.

The physical properties of the powdered extract are detailed in Table 2. The moisture content of the nine formulations ranged from 6% to 8%, with extracts showing no excessive stickiness or moisture, ensuring optimal flow characteristics. Powders with lower moisture content exhibited reduced cohesion, which positively influenced their flow properties.

The physical properties of the extract powder, including the Angle of Repose, Compressibility Index, and Hausner ratio, were used to assess and compare the flowability of the formulations. Formulations T2, T3, T7, T8, and T9 exhibited "Excellent" flowability, while the remaining formulations demonstrated "Good" flowability. These results suggest that all formulations displayed favorable flow properties, indicating their suitability for use in the production of pharmaceutical products. Formulation T9 containing 20.0% lactose monohydrate is particularly effective at absorbing viscous herbal extracts due to its hygroscopic nature and crystalline structure (Hayati et al., 2019). Its ability to efficiently bind moisture allows it to stabilize (Zeman et al., 2021) the consistency of the extracts during granulation.

Furthermore, it is essential to monitor the potential bioavailability of the active compounds and evaluate the efficacy of the granules prepared to assess their pharmacological activity.

Table 2 Physical properties of the extract powder for each formulation.

Physical properties	T1	T2	T3	T4	T5	T6	T7	T8	T9
Moisture content (%)	6.765 ±0.69	7.407 ±0.27	6.821 ±0.67	7.565 ±0.43	7.008 ±0.58	6.577 ±0.52	7.906 ±0.40	7.753 ±0.14	8.014 ±0.14
Angle of repose (θ)	35.5 ±1.03	31.5 ±1.23	31.4 ±1.56	30.2 ±2.27	32.5 ±2.65	34.0 ±1.62	29.7 ±1.25	32.6 ±1.18	29.9 ±1.56
Bulk density (g/ml)	0.580 ±0.00	0.616 ±0.00	0.657 ±0.01	0.566 ±0.01	0.573 ±0.01	0.565 ±0.01	0.608 ±0.02	0.587 ±0.01	0.578 ±0.01
Tapped density (g/ml)	0.644 ±0.01	0.656 ±0.01	0.705 ±0.02	0.641 ±0.01	0.656 ±0.01	0.643 ±0.01	0.663 ±0.03	0.618 ±0.01	0.630 ±0.02
Compressibility index (%)	11.00 ±1.01	6.00 ±0.21	7.00 ±0.51	12.00 ±1.28	13.00 ±2.70	12.00 ±2.41	8.00 ±2.98	5.00 ±1.01	8.00 ±0.13
Hausne Ratio	1.12 ±0.01	1.06 ±0.01	1.07 ±0.01	1.13 ±0.02	1.15 ±0.03	1.14 ±0.03	1.09 ±0.04	1.05 ±0.01	1.09 ±0.01

Remark: Data represent as average ± SD

5. Conclusion

From the powdered extract preparation experiment, the formulation containing 20.0% lactose monohydrate (Formulation T9) was identified as the most suitable. This formulation achieved the highest percentage yield (65%) and exhibited excellent flow properties, with moisture content maintained within an

optimal range. These favorable characteristics suggest its potential for pilot-scale production, facilitating the development of pharmaceutical products derived from herbal extracts. Additionally, this study highlights a strategy for reducing waste and promoting a more environmentally sustainable extraction and manufacturing process in the future.

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7. References

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