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Optimization of Microwave-Assisted Granules Drying for Preparation of Tomato Extract Tablets Using Wet Granulation Method

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Abstract: This work aimed to optimize tomato extract granule drying using a microwave to shorten the granule drying process by the 3^2 full factorial design. The power and time of the microwave were optimized to minimize moisture content and maximize lycopene content. Power was varied from 300 W, 450 W, and 600 W for 10 min, 15 min, and 20 min. Results showed that the optimal condition within the design space and control space was the power of 450 W for 17 min. This condition gave the moisture content of 2.09 ± 0.15% and lycopene content of 0.0798 ± 0.0031%. A low percent error of the prediction was found, indicating that the prediction by a computer program was accurate and reliable. The granules obtained from the optimal condition were further used to prepare tomato extract tablets. Three batches of 650 mg tomato extract tablets were prepared using 1500 psi compression force. All three batches had a hardness of 9.19–10.18 kP, low friability (0.026–0.033%), short disintegration time (1.44–1.70 min), and lycopene content of approximately 0.19%. This work was the first report on optimizing tomato extract granule drying using a microwave-assisted technique according to scientific novelty. In summary, microwave-assisted drying could be applied in preparing tomato extract tablets by shortening the granules drying process.

Keywords: design space, lycopene, factorial design, moisture content.

湿法制粒法制备番茄提取物片剂微波辅助干燥工艺的优化

摘要：这项工作旨在通过32全因子设计优化使用微波的番茄提取物颗粒干燥，以缩短颗粒干燥过程。微波的功率和时间经过优化，以最大限度地减少水分含量并最大限度地提高番茄红素含量。功率从300瓦、450瓦和600瓦不等，持续10分钟、15分钟和20分钟。结果表明，设计空间和控制空间内的最佳条件是功率为450瓦，持续17分钟。该条件给出了2.09±0.15%的水分含量和0.0798±0.0031%的番茄红素含量。发现预测的误差百分比很低，表明计算机程序的预测准确可靠。从最佳条件获得的颗粒进一步用于制备番茄提取物片剂。使用1500磅每平方英寸的压力制备三批650毫克番茄提取物片剂。所有三个批次的硬度均为9.19-10.18千里庞德，脆碎度低（0.026-0.033%），崩解时间短（1.44-1.70分钟），番茄红素含量约为0.19%。这项工作是根据科学新颖性使用微波辅助技术优化番茄提取物颗粒干燥的第一份报告。综上所述，微波辅助干燥可用于制备番茄提取物片，缩短颗粒干燥过程。

关键词：设计空间, 番茄红素, 因子设计, 水分含量。

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1. Introduction

Tablets are widely used in the conventional dosage form. There are three methods for preparing tablets — direct compression, wet granulation, and dry granulation. The powder with poor flowability or bulky characteristic cannot be compressed to tablets by direct compression techniques. So, the wet or dry granulation techniques are alternatives to preparing the tablets. In the case of the wet granulation method, granule preparation is an important step to promote the good flowability of the mixture. Several drying techniques are applied in drying the plant raw materials, *i.e.*, sun drying, shade drying, hot-air drying, freeze-drying, microwave drying, microwave vacuum drying, solar-assisted drying, heat pump drying, infrared drying, fluidized bed drying, etc. [1]. Hot-air drying is the most common technique used for granule drying in the pharmaceutical industry.

Microwave-assisted drying is a modern drying technique with a certain eco-friendly process. Formerly, the microwave is succeeded in applying the granule drying process. Dynamic microwave-assisted drying of granules could preserve aspirin, a moisture-sensitive non-steroidal anti-inflammatory drug, from degradation by the drying process compared with the other techniques such as hot-air drying, fluidized bed drying, and static microwave-assisted drying [2, 3]. It can be described by the shorter drying time of the granule; drug granules exposed to moisture are shorter in microwave-assisted drying compared with the other techniques, so aspirin is less degraded when drying by microwave [4]. The microwave-assisted drying technique is compared with fluidized bed drying for ibuprofen, the non-steroidal anti-inflammatory drug. It is found that microwave-assisted drying has a shorter duration time, consequently decreasing energy consumption and production cost [5, 6]. So, microwave-assisted drying could be used as an alternative method for granule drying.

Tomato (*Solanum lycopersicum* L.) extract is a lycopene-enriched source. Common varieties of tomato contain 1.60–18.46 mg lycopene/100 g edible portion [7]. Furthermore, the other plants, *i.e.*, pink guava, watermelon, pink grapefruit, papaya, persimmon, mango, wild cherry, pumpkin, sweet potato, carrot, apricot, rosehip, etc. [7, 8], also contain lycopene. Lycopene exhibits antioxidant activity, modulation of lipid metabolism, antineoplastic activity, antidiabetic activity, anti-inflammatory activity against dermatologic diseases, neuroprotective activity, bone protective activity, hepatoprotective activity, targeting reproductive disorders, etc. [7, 8].

However, lycopene could be degraded when exposed to heat, light, oxygen, metals, and oxidizing agents [9]. In addition, exposure to higher microwave power and a longer duration time of microwave irradiation could lessen its content. So, optimizing microwave-assisted drying of tomato extract and lycopene is necessary to preserve its content and activities.

This work aimed to optimize tomato extract granule drying using microwave by the 3^2 full factorial design to maximize lycopene content with suitable moisture content. Furthermore, the optimized granules were used to prepare tomato extract tablets using wet granulation methods as supplementary antioxidant products.

2. Materials and Methods

2.1. Materials

Tomato extract powder was purchased from Asian Bioplex, Chonburi, Thailand. Lycopene (purity 99.1%) was purchased from Chengdu Biopurify Phytochemicals Ltd., Sichuan, China. Polyvinyl pyrrolidone (PVP K30) (Kollidon® 30) was purchased from BASF, Ludwigshafen, Germany. Microcrystalline cellulose (Avicel® PH-101) was purchased from Fluka, Missouri, USA. Magnesium stearate was obtained from Changzhou Kaide Imp. & Exp. Co., Ltd., Changzhou, China. Talcum (Tabglide®) was purchased from Nitika Pharmaceutical Specialities Pvt. Ltd., Nagpur, India. Colloidal silicon dioxide was purchased from P.C. Drug Center, Bangkok, Thailand. Sodium starch glycolate was obtained as a gift from Onimax Co., Ltd., Bangkok, Thailand. Tetrahydrofuran was purchased from Carlo Erba Reagents S.A.S., Val de Reuil, France.

2.2. Design of Experiment of Granules Drying Using Microwave

The 3^2 full factorial design was applied for the design of the experiment of granules drying using a microwave. The design comprises two factors, *i.e.*, microwave power (300 W, 450 W, and 600 W), microwave time (10 min, 15 min, and 20 min), and two responses, *i.e.*, moisture content and lycopene content.

The master formula and working formula are shown in Table 1. The working formula was calculated for 40 tablets. First, granules of tomato extract were prepared by mixing tomato extract powder, microcrystalline cellulose, and polyvinyl pyrrolidone using mortar and pestle by the geometric dilution technique.

After the powder mixture was homogeneously mixed, 24 mL of water was added and blended with the powder mixture to produce a damp mass. Next, the damp mass was passed through a 14-mesh sieve to produce wet granules. Finally, they were spread on a pan of a microwave oven (MS23F300EEK, Samsung, Bangkok, Thailand) and dried. The microwave power and duration time used for drying granules are shown in Table 2.

Table 1 Master formula and working formula of tomato extract tablets

Ingredients	Master formula (%)	Working formula (g)	Amount per tablets (mg)	Function
Tomato extract powder	30.77	8	200	Active ingredient
Microcrystalline cellulose	59.23	15.4	385	Diluent
Polyvinyl pyrrolidone	3	0.78	19.5	Binder
Sodium starch glycolate	2	0.52	13	Disintegrant
Colloidal silicon dioxide	1	0.26	6.5	Glidant
Magnesium stearate	1	0.26	6.5	Lubricant, anti-adherent
Talcum	3	0.78	19.5	Glidant, anti-adherent
Total	100	26	650	

2.3. Determination of Moisture Content of Granules

Approximately 2 g of tomato granules were analyzed for their moisture content using moisture balance (MAC 50/NH, Radwag, Bracka, Poland). The constant temperature of 120°C was applied. The moisture content was collected when the weight change was less than 1 mg within 2 min. They were analyzed in triplicate. The average value and standard deviation (SD) were reported.

2.4. Analysis of Lycopene Content of Granules

Standard lycopene (1 mg) was dissolved in tetrahydrofuran and adjusted in a volumetric flask to 10 mL. First, a stock solution of standard lycopene in 100 µg/mL was obtained. Then, it was diluted to 1, 2, 3, 4, 5, 6, and 7 µg/mL working solutions. Next, the standard working solutions (100 µL) were pipetted to a 96-well plate (n = 3) and measured the absorbance by a microplate reader (Biorad Laboratories Inc., California, USA) at 476 nm. Tetrahydrofuran was used as a blank.

Tomato extract granules (50 mg) were dissolved in tetrahydrofuran in a 5-mL volumetric flask. They were ultrasonicated for 10 min and filtered using a nylon syringe filter with 0.45-µm pore size. The sample solutions (100 µL) were pipetted to a 96-well plate (n = 3) and measured the absorbance as the above method. In addition, the average value and SD of lycopene content were reported.

2.5. Optimization, Construction of Design Space and Control Space, and Verification of Granules Drying by Microwave

Results of moisture content and lycopene content obtained from each drying condition were analyzed by license Design-Expert® (v. 11) software. Mathematical models and actual equations were reported. The contour plots of moisture content and lycopene content were constructed. The design space was constructed in which the moisture content and lycopene content of granules were equal to or greater than hot air drying at 45°C for 6 h. Furthermore, the control space that moisture content was equal to or less than 3.5 and lycopene content was equal to or higher than 0.08 was also produced. The optimal condition within the control space was selected to prepare the tomato extract granules for the verification step. The experimental value obtained from the optimal condition of granules drying was compared with the predicted value to verify the accuracy and reliability of the prediction by computer software. Percent error was calculated as Equation 1.

$$Error\ (\%) = \left(\frac{Experimental\ value - Predicted\ value}{Experimental\ value}\right) \times 100 \quad (1)$$

2.6. Preparation of Tomato Extract Tablets

Premix consisted of sodium starch glycolate, colloidal silicon dioxide, magnesium stearate, and talcum. Then, it was mixed for 3 min with the tomato extract granules, which were passed through a 20-mesh sieve. The obtained mixture was weighed for 600 mg and compressed using an in-house assembled hydraulic press connected with a pressure gauge. Each tablet was compressed using 1500 psi force and maintained for 10 s.

2.7. Evaluation of Physicochemical Properties of Tomato Extract Tablets

The tablets were evaluated for their physicochemical properties in six topics, *i.e.*, average weight, weight variation, thickness, diameter, hardness, friability, disintegration time, and lycopene content.

Individual weight was measured according to average weight by analytical balance (Entris224i-1S, Sartorius AG, Göttingen, Germany) (n = 20). The average value and SD were reported. Weight variation was calculated by comparing the difference between individual weight (W_i) and average weight (W_a) as Equation 2.

$$Weight\ variation\ (\%) = \left(\frac{W_i - W_a}{W_a}\right) \times 100 \quad (2)$$

Thickness and diameter were measured using a thickness gauge (n = 20). Hardness was measured by a hardness tester (TBH 220 TD, Erweka GmbH, Heusenstamm, Germany) (n = 10). Eleven tablets were dust removed and then weighed (W_1) using an analytical balance. They have tested the friability using a friability tester (K.S.L. Engineering Co. Ltd., Bangkok, Thailand).

The drum of the friability tester was rotated at 25 rpm for 4 min. Then, the tablets were removed from the drum, dust removed, and weighed (W_2). The friability was calculated using Equation 3.

$$Friability\ (\%) = \left(\frac{W_1 - W_2}{W_1}\right) \times 100 \quad (3)$$

Disintegration time was evaluated using a disintegration tester. Water at 37°C ± 0.5°C was used as a disintegration medium. Six tablets were tested by measuring individual disintegration time. The average

value and SD were reported.

Ten tablets were ground to a fine powder using a mortar and pestle. The 600 mg powder was weighed, filled into a 10-mL volumetric flask (n = 3), diluted with tetrahydrofuran, and ultrasonicated for 10 min. They filtered and analyzed lycopene content using the above method.

3. Results and Discussion

3.1. Granule Drying Using Microwave

Moisture content and lycopene content of dried granules containing tomato extract powder, microcrystalline cellulose, and polyvinyl pyrrolidone are shown in Table 2. The highest and the lowest moisture contents were found in Condition 1 and Condition 9, respectively. At the same time, the highest and the lowest lycopene contents were found in Condition 5 and Condition 1, respectively. Moisture and lycopene contents were fitted to two-factor interaction (2FI) and quadratic mathematical models. Furthermore, the actual equations are shown in Table 3.

Table 2 Factors and responses of 3^2 full factorial design

Conditions	Factors		Responses	
	X_1	X_2	Y_1	Y_2
	Power (W)	Time (min)	Moisture content (%)	Lycopene content (%)
1	300	10	8.44 ± 0.46	0.0585 ± 0.0025
2	300	15	3.61 ± 1.08	0.0665 ± 0.0034
3	300	20	1.93 ± 0.13	0.0674 ± 0.0068
4	450	10	2.53 ± 0.17	0.0776 ± 0.0037
5	450	15	1.59 ± 0.17	0.0860 ± 0.0071
6	450	20	1.43 ± 0.17	0.0781 ± 0.0035
7	600	10	1.86 ± 0.47	0.0698 ± 0.0017
8	600	15	1.54 ± 0.10	0.0712 ± 0.0027
9	600	20	1.22 ± 0.09	0.0702 ± 0.0030

Table 3 Mathematical models and actual equations of moisture content and lycopene content

Responses	Mathematical models	Actual equations
Moisture content (Y_1, %)	2FI*	$Y_1 = 24.6958 - 0.0398X_1 - 1.1555X_2 + 0.0020X_1X_2$
Lycopene content (Y_2, %)	Quadratic	$Y_2 = -0.1087 + 0.0006X_1 + 0.0068X_2 - (2.8632 \times 10^{-6})X_1X_2 - (5.9228 \times 10^{-7})X_1^2 - 0.0002X_2^2$

* 2FI is two-factor interaction.

Contour plots of moisture content and lycopene content are shown in Fig. 1. According to moisture content, increasing microwave power and time and the interaction between microwave power and time decreased its value. In the case of lycopene content, the optimal condition was observed. Increasing microwave power from 300 W to 450 W increased lycopene content while increasing microwave power from 450 W to 600 W decreased lycopene content. At 450 W, increasing time from 10 min to 15 min increased lycopene content, while increasing time from 15 min to 20 min decreased lycopene content.

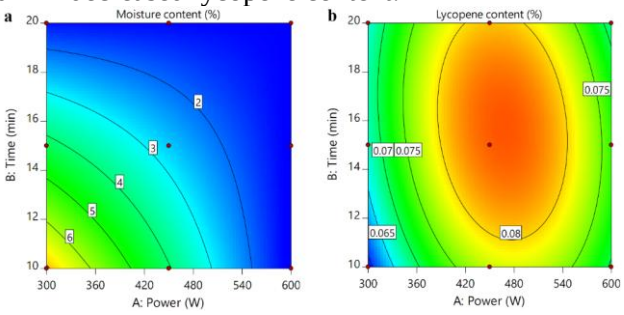

Fig. 1 Contour plots of (a) moisture content and (b) lycopene content of granule drying by microwave

Results in the present work had a similar tendency to the previous works. Microwave power is a key factor that affects moisture removal under microwave application [10]. Increasing microwave power increased the drying rate constant. Furthermore, increasing microwave time also decreased the moisture content of the materials, indicating the moisture content was decreased when microwave power and microwave time increased [11]. According to chemical analysis, for thermo-stable compounds, increasing microwave power or time increases active compounds rather than degradation. For example, increasing microwave time from 20 to 60 min at 630 W decreased the water content of sliced turmeric while increasing total volatile oil content and total curcuminoid content due to water content decreased [12]. Moreover, microwave heating enhanced the biological activity of the plant — microwave heating enhanced the antioxidant activity of tomatoes by enhancing polyphenol and flavonoid contents [13]. However, microwave-assisted drying could alter the profiles of some thermo-labile compounds, such as volatile substances [14].

In the case of the thermo-labile compound — lycopene, increasing microwave power and time could decrease its content. Furthermore, lycopene could also be degraded when exposed to light, oxygen, metals, and oxidizing agents. Therefore, it is recommended that care should be taken to minimize the exposure to oxidizing agents, especially when intense heat (higher than 100°C) is applied [9]. The most stable form of lycopene is the all-*trans* isomer, accounting for approximately 95% of the lycopene present in red tomatoes [15]. However, microwave heating affected lycopene degradation and isomerization of lycopene from all-*trans* to *cis*-lycopene [16]. At 100°C and 120°C treatments, all-*trans* to *cis*-lycopene isomerization has rapidly occurred. Consequently, the *cis*-form is unstable and rapidly degraded [17].

3.2. Design Space, Control Space, and Optimal Condition

Design space provided the moisture and lycopene

contents of dried granules obtained from microwave-assisted drying were equal to or greater than drying by the hot air oven at 45°C for 6 h — moisture content of 3.82% lycopene content of 0.0715%, is shown in Fig. 2a. In addition, the control space, which narrowed the design space, was produced using the moisture content of 3.5% and lycopene content of 0.08%, ensuring the moisture and lycopene content of dried granules obtained from microwave-assisted drying were superior the hot air drying. The control space is shown in Fig. 2b.

The optimal condition for microwave-assisted drying of granules inside the control space was microwave power of 450 W for 17 min. This optimal condition was different from the previous work; optimal microwave-vacuum drying of sliced tomato gave the highest lycopene content at a microwave power of 700 W [18]. This occurrence might associate with the different raw materials — sliced tomato vs. tomato extract granules.

The optimal condition: 450 W for 17 min, was used to verify the prediction accuracy by the computer software: Design-Expert® (v. 11). Verification data are shown in Table 4. They were found that the percent errors of both moisture content and lycopene content were lower than 5%. Furthermore, the experimental values of moisture content and lycopene content were within a 95% confidence interval, indicating that the prediction was accurate.

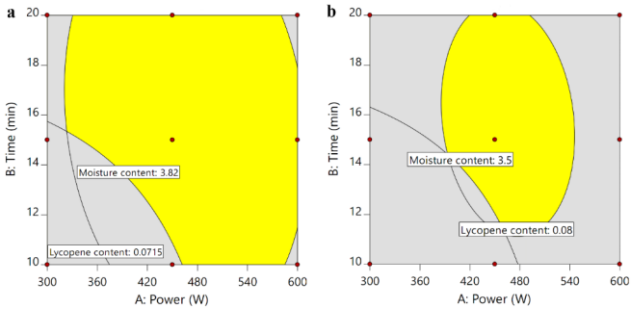

Fig. 2 Overlay plots demonstrated (a) design space that moisture content and lycopene content was equal to hot air drying (moisture content was equal to or less than 3.82% and lycopene content was equal to or higher than 0.0715%) and (b) control space that moisture content was equal to or less than 3.5% and lycopene content was equal to or higher than 0.08%

Table 4 Verification results of moisture content and lycopene content of granules drying by microwave at 450 W for 17 min

Responses	Predicted values	Experimental values (n = 3)	Error (%)	95% CI	
				Lower	Upper
Moisture content (%)	2.13	2.09 ± 0.15	–1.91	1.01	3.26
Lycopene content (%)	0.0834	0.0798 ± 0.0031	–4.51	0.0773	0.0895

These results indicated that microwave-assisted drying of tomato extract granules is equal to or better than hot air drying in terms of moisture content and lycopene content. Hence, microwave-assisted drying is an interesting technique for drying granules to prepare supplementary products in tablet form due to rapid heating rates and short processing time, saving energy. Furthermore, it provides desired product properties and does not generate secondary waste in an eco-friendly manner [19].

3.3. Physicochemical Properties of Tomato Extract Tablets Prepared by Wet Granulation Method

Tablets with low friability and suitable hardness became a good performance indicating that tablets had sufficient physical strength and did not lose components due to abrasion, friction, or mechanical shock [20]. Furthermore, tablets with a short disintegration time could provide rapid drug absorption and drug acting compared with the tablets with a longer disintegration time [21]. The dried granules obtained from microwave-assisted drying were mixed with the premix of sodium starch glycolate, colloidal silicon dioxide, magnesium stearate, and talcum. Finally, the obtained granule mixture was compressed into a tablet in three batches. Their physicochemical properties, including average weight, thickness, diameter, hardness, friability, disintegration time, and lycopene content, are shown in Table 5. Results showed that all batches had similar physicochemical properties. Furthermore, no tablet had an individual weight outside the 5% of average weight, indicating that the weight variation was acceptable. In addition, the tablets with low friability and short disintegration time were achieved.

Table 5 Physicochemical properties of tomato extract tablets prepared by wet granulation method which granules were dried by optimal microwave condition

Parameters	Batch 1	Batch 2	Batch 3
Average weight (mg)	657.69 ± 0.96	657.77 ± 1.26	654.87 ± 2.04
Thickness (mm)	4.63 ± 0.02	4.67 ± 0.02	4.66 ± 0.02
Diameter (mm)	12.83 ± 0.01	12.85 ± 0.02	12.84 ± 0.02
Hardness (kP)	10.18 ± 1.28	9.63 ± 0.43	9.19 ± 0.38
Friability (%)	0.033	0.026	0.027
Disintegration time (min)	1.70 ± 0.33	1.44 ± 0.27	1.47 ± 0.17
Lycopene content (mg)	0.1867 ± 0.0230	0.1897 ± 0.0263	0.1853 ± 0.0303

4. Conclusion

The tomato extracted granule drying conditions, i.e., microwave power and microwave time, were optimized using the 3^2 full factorial design. The optimal condition gave the low moisture content, and high lycopene content was microwave power of 450 W for 17 min. This condition gave the inferior moisture content and superior lycopene content with a shorter drying time compared to drying in a hot air oven. The tomato extract tablets prepared from granules dried by microwave-assisted drying technique had desired properties with low friability and short disintegration time. In summary, microwave-assisted drying could be an alternative technique for drying tomato extract granules.

Furthermore, the obtained granules were suitable for

preparing tomato extract tablets. The novelty of this work was the first report on the optimization of tomato extract granule drying using a microwave-assisted technique. The limitation of this work was the lack of stability data of tomato extract tablets; however, it will be evaluated in future work.

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