

Optimization of Spray Drying Without Drying Carriers from *Rehmannia glutinosa* L. Root and *Coix lachryma-jobi* L. Trunk

Linh Tuyen Thi Nguyen¹ , Truc Trung Tran², Ngoc-Van Thi Nguyen^{1,*} 

¹ College of Pharmacy, Can Tho University of Medicine and Pharmacy, 179 Nguyen Van Cu Street, Can Tho City, Vietnam; nltuyen@ctump.edu.vn (L.T.T.N.);

² Imexpharm Corporation, Vietnam; trucmouse46@gmail.com (T.T.T.);

* Correspondence: nguyenthingocvanct@gmail.com (N-V.T.N.);

Scopus Author ID: 55420442800

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Abstract: Spray drying is a well-known method for preparing dried extracts from herbs. *Rehmannia glutinosa* root and *Coix lachryma-jobi* trunk are widely researched especially for diabetes treatment. The optimization of extraction conditions, namely the alcohol concentration, treated herbs and solvent ratio, extraction temperature, and time. The extracts are dried by Labplant machine with two specifications (drying inlet air temperature and flow- rate). *Rehmannia glutinosa* root is extracted by refluxing method for 60 minutes with 40% alcohol, the ratio of treated herbs and solvent was 1:3, at 80°C by three times extract and its dried extract is prepared at a drying inlet air temperature of 90°C, 8 cycles per minute. *Coix lachryma-jobi* trunk is also extracted by the same method for 60 minutes with 80% alcohol, the ratio of treated herbs and solvent was 1:6, extraction temperature and time are respective 80°C and two times, spray drying conditions were 80°C (drying inlet air temperature) and 4 cycles per minute (flow- rate). The obtained dried extracts of *Rehmannia glutinosa* root and *Coix lachryma-jobi* trunk have average moisture contents of 4.52% and 3.84%, respectively. Therefore, this study is a novel approach to significantly decreasing the amount of carriers to reduce research's funds and saving dried extracts' price.

Keywords: Spray drying without drying carriers, extraction optimization, *Rehmannia glutinosa* L. root, *Coix Lachryma- Jobi* L. trunk.

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1. Introduction

Rehmannia glutinosa (Gaertn.) Libosch. ex Steud - a widely used herb of traditional medicine belongs to the *Scrophulariaceae* family. In Vietnam, this precious plant has grown a lot in the Northern. *Rehmannia glutinosa* root has many bioactive compounds such as oligosaccharide, catalpol, rehmannioside D, which are often clinically used to ameliorate symptoms of inflammation, cardiovascular diseases, and especially for patients who have diabetes [1-4]. Zhang *et al.* investigated and showed a high hypoglycemic effect on diabetic rats (glucose and adrenaline-induced; and alloxan-induced) of water and stachyose extract from this herb [5].

Coix lachryma jobi L., also called adlay, is a distant relative of maize in the Maydace tribe of the grass family, Poaceae or Gramineae. This herb has been feral in many periods and recently in Vietnam, and has just been planted in some provinces like Nghe An, Thanh Hoa; Job's tear has been used for a long time in traditional medicine and as a nourishing cereal by adding

its flour or whole grain in soups and broths. Recent studies proved that *Coix* seed extract protects against oxidative stress, exhibits anti-inflammatory, and improves lipid metabolism from tumor stimulating compounds [6-9]. Some phytosterols such as stigmast-4-en-3-on, stigmasterol, β -sitosterol, coixans A, B, and C are reported to be hypoglycemic agents [10-12].

Extraction is a process in which soluble plant chemical constituents (including those which have therapeutic activity) are separated from insoluble plant metabolites and cellular matrix, using selective solvent (which is sometimes called menstruum). The purpose of extracting herbal material is to eliminate unwanted materials and concentrate other chemical constituents in a soluble form. Herbal extracts include liquid (fluid) extracts, soft extracts, oleoresins, dry extracts, and others [13]. Most of these requirements can be accepted by a spray-dried powder extract. One of the most important characteristics of powders is the high content of sensitive and health-beneficial compounds. Spray-dried extracts can easily be transported, handled, and reduced in bulk because of their high stability. All these characteristics make more studies interested in using these dry powders [14-16].

Spray drying is a well-known particle production method that transforms a fluid material into dried particles, taking advantage of a gaseous hot drying medium. The spray-drying mechanism uses a heated atmosphere to eliminate moisture from the pre-product. The procedure is described by three major stages, including atomization, droplet-to-particle transformation, and collection [17]. Spray drying is widely applied in some sectors of industry, including pharmaceutical, chemical, and food. Several biological and thermal-sensitive materials, liquid materials (milk, fruit juices and pulps, herbal extracts, enzymes, essential oils, aromas), and a lot of medicines have been dried by this process [18-20]. Nowadays, the preparation of dried extracts using the spray-drying method without drying carriers is more being researched and applied. Dried extracts prepared by this method achieve physical and chemical stability, reduce the risk of infection, high solubility, and outstanding activity content compared to other drying methods [21,22].

In addition, there are no published documents on the preparation of dried extracts from *Rehmannia glutinosa* root and *Coix lachryma-jobi* trunk worldwide. Therefore, the purpose of this study is finding the conditions for extracting and preparing a dried extract from *Rehmannia glutinosa* root and *Coix lachryma-jobi* trunk by spray drying method without drying carriers.

2. Materials and Methods

2.1. Materials.

Ethanol was purchased from Merck Company (Darmstat, Germany); distilled water met analytical standards. *Rehmannia glutinosa* root, *Coix lachryma-jobi* trunk is provided from Can Tho Hospital of Traditional Medicine and Vinh Long province, respectively, in the South of Vietnam, which are ground into raw powder. These powders contain no more than 9 percent of water and meet the testing standards according to Pharmacopoeia of the People's Republic of China (2005) Vol 1.

2.2. Instrumentation.

Spray drying without drying carriers method is carried out using SD-06AG Spray dryer SD-06AG (Labplant, UK).

2.3. Sample preparation.

In our study, we use the reflux and spray drying without carrier method to investigate extraction conditions as well as the Spray dryer parameters to get the end optimization of the extraction process. Those processes are described below:

2.3.1. Reflux extraction.

There are 4 factors that have been chosen to investigate: alcohol concentrations (%), the ratio of treated herbs and solvent, temperature (°C) and extraction times.

2.3.1.1. *Rehmannia glutinosa* root.

Approximately 80g of *Rehmannia glutinosa* root is weighed, used alcohol solvents at different concentrations (30%, 40%, 50%, 60%), the ratio of treated herbs and the solvent is 1:4, at 60°C by two times extract, refluxing for 60 minutes, determining the appropriate concentrations. Then, surveying the ratio of treated herbs and solvent (1:2, 1:3, 1:4, 1:5) with the suitable alcohol concentration, researching with temperature (60°C, 70°C, 80°C, 90°C) and extraction times (2, 3, 4), respectively.

2.3.1.2. *Coix lachryma-jobi* trunk.

Restarting the above survey for *Coix lachryma-jobi* trunk with a different survey value: quantity of medicinal herbs tested is 100g, the determination alcohol concentrations is 60%, 70%, 80%, 90%, respectively, with the ratio of treated herbs and the solvent is 1:6, at 60°C by two times extract, also using reflux extraction for 60 minutes; the surveyed ratio of treated herbs and the solvent is 1:5, 1:6, 1:7, 1:8; at 60°C, 70°C, 80°C, 90°C by 2; 3; 4 times for each experiment.

Table 1. Extraction conditions.

Run	<i>Rehmannia glutinosa</i>				<i>Coix lachryma-jobi</i>			
	X1 (%)	X2	X3 (°C)	X4	X'1 (%)	X'2	X'3 (°C)	X'4
1	30	1:4	60	2	60	1:6	60	2
2	40	1:4	60	2	70	1:6	60	2
3	50	1:4	60	2	80	1:6	60	2
4	60	1:4	60	2	90	1:6	60	2
5	X _{1T}	1:2	60	2	X'1 _T	1:5	60	2
6	X _{1T}	1:3	60	2	X'1 _T	1:7	60	2
7	X _{1T}	1:5	60	2	X'1 _T	1:8	60	2
8	X _{1T}	X _{2T}	70	2	X'1 _T	X'2 _T	70	2
9	X _{1T}	X _{2T}	80	2	X'1 _T	X'2 _T	80	2
10	X _{1T}	X _{2T}	90	2	X'1 _T	X'2 _T	90	2
11	X _{1T}	X _{2T}	X _{3T}	3	X'1 _T	X'2 _T	X'3 _T	3
12	X _{1T}	X _{2T}	X _{3T}	4	X'1 _T	X'2 _T	X'3 _T	4

*X1, X'1: the concentration of alcohol; X2, X'2: the ratio of treated herbs and solvent; X3, X'3: extraction temperature; X4, X'4: extraction times; X_{1T}, X'1_T: the optimal concentration of alcohol; X_{2T}, X'2_T: the optimal ratio of treated herbs and solvent; X_{3T}, X'3_T: optimal extraction temperature.

2.3.2. The spray drying without carrier method.

Preparing *Rehmannia glutinosa* root and *Coix lachryma-jobi* trunk extracts according to the above determining conditions, then applying the spray drying without drying carrier method with all obtaining extracts. There are two surveying parameters in this study: temperature (°C) and flow rate (rpm).

Rehmannia glutinosa root extract uses 2.5 bar inlet air pressure with temperature (°C): 80, 85, 90, 95; Flow rate (rpm): 6, 8, 10.

Coix lachryma-jobi trunk extract uses 1.0 bar for inlet air pressure with temperature (°C): 70, 75, 80; Flow rate (rpm): 4, 6, 8.

Table 2. Spray drying conditions*.

Run	Rehmannia glutinosa		Coix lachryma-jobi	
	Temp. (°C)	Flow rate (rpm)	Temp. (°C)	Flow rate (rpm)
1	80	6	70	4
2	80	8	70	6
3	80	10	70	8
4	85	6	75	4
5	85	8	75	6
6	85	10	75	8
7	90	6	80	4
8	90	8	80	6
9	90	10	80	8
10	95	6		
11	95	8		
12	95	10		

*Inlet air pressure (bar): 2,5 (*Rehmannia glutinosa*); 1,0 (*Coix lachryma-jobi*).

2.4. Response variables analysis.

The obtained dried extract has the highest extraction efficiency (Y_1) and the lowest moisture (Y_2) and does not exceed 5%. About appearance, the dried extract has little adhesion, easy to collect.

The efficiency of dried extract (%) is calculated by the formula:

$$Y_1 = \frac{b(100 - Y_2)}{a(100 - h_a)} \times 100$$

Includes:

a: quantity of medicinal herbs tested (g),

b: quantity of dried extract (g),

h_a : moisture of medicinal herbs (%),

Y_2 : moisture content of dried extract (%).

3. Results and Discussion

3.1. Optimization of the extraction conditions.

The choice of the optimal reflux extraction conditions is performed by highest efficiency after the survey. The result shows that *Rehmannia glutinosa* root reaches the highest level in efficiency when using 40% alcohol, in the ratio of 1:3 (herbs: solvent), at 80°C by three times extract. (*Rehmannia glutinosa*. Run 11. Table 3) While for *Coix lachryma-jobi* trunk, 80% alcohol, 1:6 ratio of treated herbs and solvent, 80°C and two times extraction will get its peak. (*Coix lachryma-jobi*. Run 9. Table 3).

3.1.1. Solvent Investigation.

This is the first examining factor because it affects the most extraction efficiency and plays a role in dissolving substances in medicinal herbs. This chosen factor is bases on the polarity of the compounds contained in the medicinal plant and the properties of the solvent.

Rehmannia glutinosa's compounds are easy to dissolve in water and dilute alcohol [23,24]. However, the aqueous solvent also dissolves many impurities, making the active ingredient preserve difficultly and unstable, while alcohol solvent dissolves more active ingredients and fewer impurities [23], so the alcohol-water mixture is selected. By using 40% alcohol solvent, this experiment gets the highest efficiency after calculating. Meanwhile, Phytosterol in *Coix lachryma-jobi* is less polar and oily, so the solvent is alcohol with a higher rate of concentrations 80%, which also has the biggest Y1's value.

3.1.2. The ratio of treated herbs and solvent investigation.

The treated herbs and solvent ratio is appropriately selected to match the extraction condition and save time and energy consumption for further drying. Although the values of moisture of medicinal herbs are in the near middle rate, the chosen ratio for each plant still achieves the standard of efficiency.

3.1.3. The temperature investigation.

High temperature can affect the increased solubility and diffusion rate and destroy the organization of medicinal cells to increase the efficiency of the extraction process. However, the excessive increase of temperature can raise impurities in the extract, destroy the active ingredients. The chosen temperatures are under the decomposition temperature and the experiment with the highest efficiency in this survey.

3.1.4. The extraction times investigation.

The herbal experiments in this study prove that not always the more times we extract, the more efficiency we get. Consequently, surveying suitable extraction times is insurance for the maximum concentration of active ingredients in herbal extraction.

Table 3. Extraction optimization.

	Run	X ₁	X ₂	X ₃	X ₄	a	b	Y ₁	Y ₂
Rehmannia glutinosa	1	30	1:4	60	2	80.12	8.65	11.35	4.57
	2	40	1:4	60	2	79.95	10.25	13.54	4.10
	3	50	1:4	60	2	80.43	9.28	12.21	3.93
	4	60	1:4	60	2	80.24	8.94	11.78	4.00
	5	40	1:2	60	2	79.93	8.94	11.83	3.93
	6	40	1:3	60	2	80.18	10.52	13.86	4.08
	7	40	1:5	60	2	80.06	10.31	13.56	4.37
	8	40	1:3	70	2	80.27	11.90	15.63	4.23
	9	40	1:3	80	2	80.21	13.86	18.24	4.13
	10	40	1:3	90	2	80.15	14.25	18.76	4.17
	11	40	1:3	80	3	79.83	15.58	20.60	4.13
	12	40	1:3	80	4	80.44	15.91	20.87	4.17
Coix lachryma-jobi	1	60	1:6	60	2	10.02	10.21	10.59	4.15
	2	70	1:6	60	2	99.81	10.91	11.35	4.07
	3	80	1:6	60	2	100.03	15.72	16.32	4.08
	4	90	1:6	60	2	100.22	15.04	15.55	4.24
	5	80	1:5	60	2	99.80	14.20	14.78	4.05
	6	80	1:7	60	2	100.00	13.75	14.26	4.18
	7	80	1:8	60	2	99.98	13.87	14.41	4.00
	8	80	1:6	70	2	100.08	18.98	19.71	3.83
	9	80	1:6	80	2	100.13	22.35	23.19	4.02
	10	80	1:6	90	2	99.79	20.41	21.23	4.12
	11	80	1:6	80	3	100.24	19.40	20.02	4.44
	12	80	1:6	80	4	100.01	19.93	20.63	4.35

3.2. Optimization the parameter of Spray dryer.

The chosen runs are based on the efficiency as well as the average moisture contents and the sense of the product.

Rehmannia glutinosa root dried extract is prepared by spray-drying without drying carriers with drying inlet air temperature of 90°C and flow- rate is 8 rpm (*Rehmannia glutinosa*. Run 8. Table 4.) while that conditions of *Coix lachryma-jobi* trunk are 80°C (drying inlet air temperature) and 4 rpm (flow- rate) (*Coix lachryma- jobi*. Run 7. Table 4).

The obtained dried extracts of *Rehmannia glutinosa* root and *Coix lachryma-jobi* trunk have average moisture contents of 4.52%, 3.84%, respectively.

The inlet air temperature and flow rate greatly affect the volume and moisture content of the product after spray drying. The drying air temperature is low, or the flow- rate is too fast causes the amount of undried raw materials with the over-standard moisture content after drying. Consequently, the powder is very sticking to the device and reduces the total drying powder. Conversely, too high a temperature can degrade the active ingredient in medicinal herbs, while a too slow flow rate can take more time and energy for extraction [25-28].

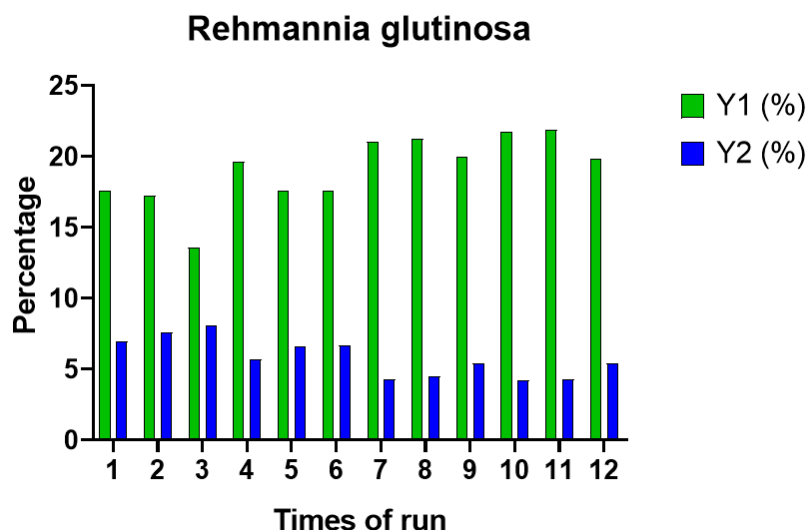


Figure 1. Extraction efficiency (Y1) and Average moisture contents (Y2) of *Rehmannia glutinosa*.

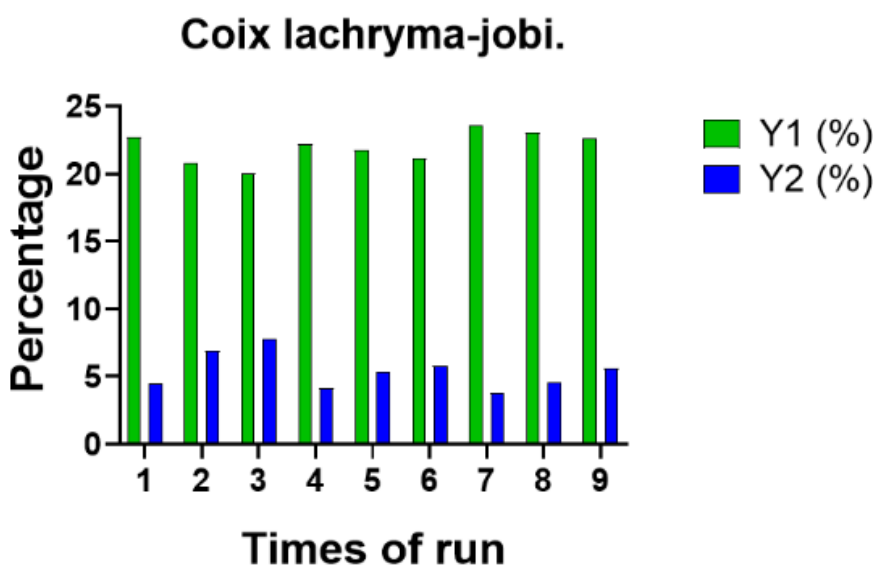


Figure 2. Extraction efficiency (Y1) and Average moisture contents (Y2) of *Coix lachryma-jobi*.

	Run	Temp. (°C)	Flow rate (rpm)	a (g)	b (g)	Y1	Y2	Appearance
Rehmannia glutinosa	1	80	6	79.84	13.68	17.56	6.95	(-)
	2	80	8	79.62	13.48	17.23	7.60	(-)
	3	80	10	79.98	10.74	13.59	8.12	(-)
	4	85	6	79.55	15.06	19.66	5.71	(-)
	5	85	8	80.04	13.66	17.56	6.58	(-)
	6	85	10	80.41	13.75	17.57	6.68	(-)
	7	90	6	79.99	15.96	21.02	4.32	(+)
	8	90	8	79.57	16.09	21.27	4.52	(+)
	9	90	10	80.05	15.38	20.01	5.43	(-)
	10	95	6	79.89	16.50	21.78	4.23	(+)
	11	95	8	79.98	16.61	21.89	4.30	(+)
	12	95	10	80.15	15.25	19.82	5.41	(-)
Coix lachryma-jobi	1	70	4	100.09	21.96	22.69	4.47	(+)
	2	70	6	99.91	20.67	20.84	6.93	(-)
	3	70	8	99.97	20.05	20.01	7.82	(-)
	4	75	4	99.94	21.40	22.21	4.16	(+)
	5	75	6	99.76	21.20	21.78	5.32	(-)
	6	75	8	99.95	20.78	21.20	5.81	(-)
	7	80	4	99.98	22.69	23.62	3.84	(+)
	8	80	6	100.05	22.34	23.07	4.55	(+)
	9	80	8	100.01	22.20	22.67	5.65	(-)

Table 4. Optimization of spray drying conditions.

*Inlet air pressure (bar): 2,5 (*Rehmannia glutinosa*); 1,0 (*Coix lachryma-jobi*).

Sense: (-): easy to moisture, clumping or sticking much on the device, can not get ;

(+): dry tall, easy to collect, light brown color.

Overall, *Rehmannia glutinosa* root and *Coix lachryma-jobi* trunk are extracted by refluxing method (for 60 minutes) and spray drying without drying carriers method. To extract *Rehmannia glutinosa* root, 40% alcohol is used at 80°C by three extracts, with the 1:3 ratio of treated herbs and solvent; spray-dried parameters are 90°C (inlet air temperature) and 8 rpm (flow-rate). *Coix lachryma-jobi* trunk is extracted with 80% alcohol. The ratio of treated herbs and solvent, extraction temperature, and time are respective 1:6, 80°C and two times, sprayed at 80°C (inlet air temperature) and 4 rpm (flow-rate). The obtained dried extracts of *Rehmannia glutinosa* root and *Coix lachryma-jobi* trunk have average moisture contents: 4.52% and 3.84%, respectively.

4. Conclusions

This study gives an economic optimization extraction. No using carrier in the spray-dried process saves expenditure's fund for that compound, leading to the reducing price of dried extract. Moreover, it can open more future research in combining two potential herb- extracts in Diabetes's treatment.

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Conflicts of Interest

The authors declare no conflict of interest.

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