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SPRAY DRYING INVESTIGATIONS ON MEDICINAL PLANT BASED PHARMACEUTICAL PRODUCTS

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Abstract

In the paper we report about spray drying investigations on medicinal herb extracts. We investigated the production technologies of up-to-date pharmaceutical products and natural raw materials of drugs. In this paper we report in detail about spray drying investigation of some medicinal plants and about how to define the operational features of these products. In our tests we could find that the extract of camomile, rose hip and lime blossom can be processed well by spray drying. We worked out the main operational features to be applied when using spray drying under industrial circumstances.

Keywords: spray drying, processing of medicinal plants, production technology of pharmaceutical products.

Introduction

The demand for different pharmaceutical products and raw material of drugs gained from medicinal plants has considerably increased in the recent years.

The active principles of medicinal plants are utilized mainly by consuming or further processing of the tinctures and solutions or (in other words) extracts gained from the plant with different methods. The use of the extract in the solving material (which is alcohol in most cases) is not convenient. In some cases the extract must be consumed or processed within a short time. The presence of the solving material may cause difficulties for the further processing. The stable presence of the active principles in a sufficient concentration can be assured only with difficulties or not at all. Especially for water dissolvent extracts the danger of becoming infected by microorganisms is serious (KEDVESSY, 1981).

In order to overcome these problems the drying of extracts is successfully used. During this procedure the dissolvent will be removed by drying and an extract which contains most of the active ingredients will be gained. The most suitable method to dry the solution is to apply spray drying.

The Advantages of Spray Drying when Drying Herbal Extracts

From the intensive drying technologies which dry the materials in a dispergated condition, spray drying is one of the most widely applied technologies used to dry drugs and herbal extracts.

The short substance of this technology is that the solution will be atomized into very small droplets by the means of a proper spraying device and the moisture will be evaporated from the drops moving very fast in the drying chamber during a very short time.

This procedure has more advantages and the most important of these are:

- the large surface obtained by the spraying assures pleasant conditions for the drying process;
- the technology is not very sensitive concerning the substance of the material, so that solutions, emulsions and pastes can be dried with this technology as well;
- the short drying time allows the drying of heat sensitive materials as well;
- the short time of staying and the mild drying may allow to save most of the active ingredients;
- it assures continuous operating conditions (continuous inlet and outlet in powder-form product).

For the drying circumstances the relative motion of the sprayed drops, the input conditions and the geometrical form of the drying chamber are important.

During the process of spray drying some very complicated physical processes take place. The sizes of the droplets coming into being in the spraying device are not equal in size but a very characteristic distribution of size is shown.

The size distribution of the drops may have a major influence on the drying process. The droplets of small size may dry out very close to the atomizing nozzle, they may saturate the air with moisture and may even cause the bigger drops to get more moisturized. At the same time it may happen that the drops of big size do not dry to the necessary extent while reaching the bottom of the drying chamber.

We can investigate the procedure of spray drying by measuring and by a computer program based on a mathematical model.

The results of the simulation confirm our previously described considerations which make spray drying so much suitable for processing of medicinal herb extracts (TOPÁR, 1980).

In the recent period of time a co-operation has been built up between the Research Institute for Medicinal Plants Corp. and the Department of Chemical and Food Engineering in working out the manufacturing technology of herb-based pharmaceutical products. Our Department tested mainly the possibilities of drying of the tinctures gained by extraction and worked out the drying technology. We are going to assume the results of this work in the following pages.

Spray Drying Experiences

We made drying technology tests on the extracts produced in the Research Institute for Medicinal Plants in the RSZL–10 type spray drying device of the semi-industrial laboratory of our Department.

The sketch of the device is shown in *Fig. 1.* The main parts of the device are: the drying chamber, the atomizing nozzle, the fan, the electrically heated heat exchanger, the cyclone and the control desk of the device.

The most important characteristics of the laboratory spray dryer are:

inner diameter of the drying chamber:	1200	mm
volume of the drying chamber:	1.2	m^3
diameter of the spraying disc:	78	mm
r.p.m. of the spraying disc:	36000	r.p.m.

The maximal nominal moisture evaporating capacity of the device is 10 kg/h. The inlet environmental air will be heated by a heat exchanger, its built-in power is 16 kW. The heating capacity can be adjusted step by step, in 16 steps.

The spray drying tests were made through on this device as follows:

The herbal extract supplied by our consigner was fed into the device from the tank (8) by a peristaltic pump (7). The speed of the feed-in could be changed continuously by changing the revolution of the pump. The mass flow of the feeding-in could be determined by measuring the starting and the rest mass and the operation time.

The inlet solution came to the spraying disc (2) that was revolving with 36000 r.p.m. and the solution fell here apart into droplets. The droplets met the air coming from the heat exchanger (4) in the drying chamber (1). Moving in the drying chamber, getting into touch with the air, the droplets dry out and leave together with the air to the powder separating cyclone (5). In the cyclone the powder will be separated from the air and the dried product comes into the bin at the bottom of the cyclone. The drying air — the moisture content of which grew during the procedure — will be exhausted through the outlet nozzle of the cyclone to the open

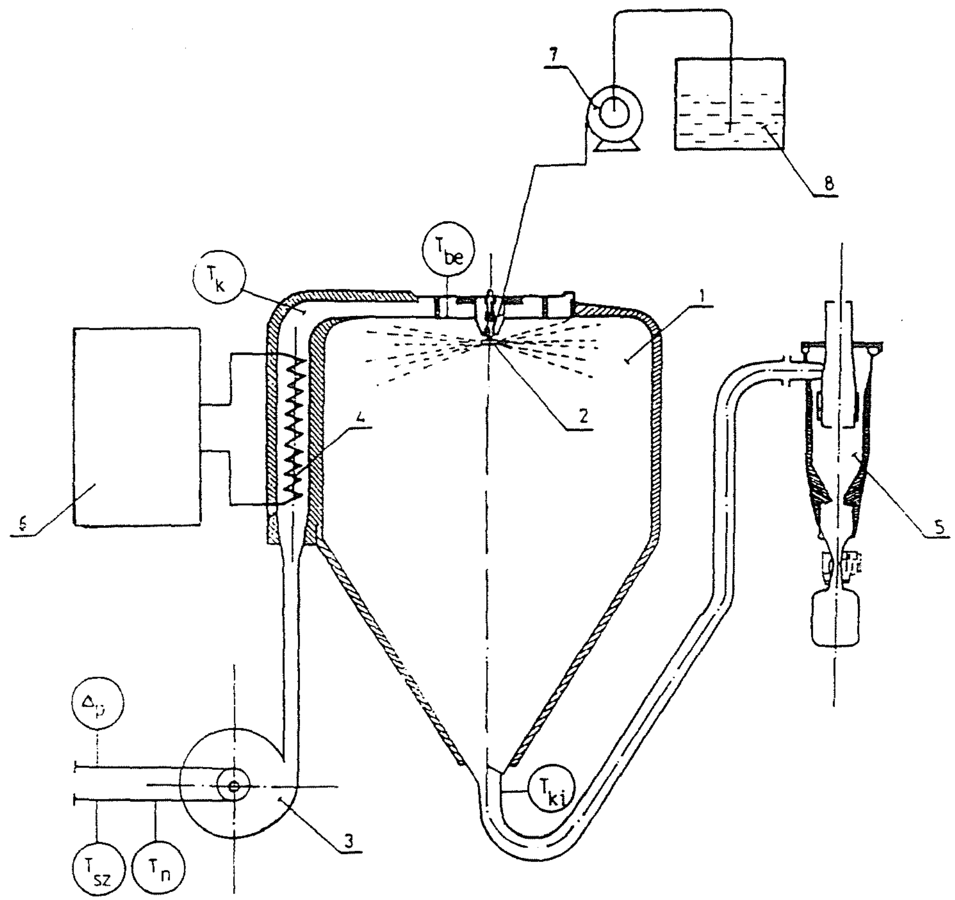

Fig. 1. Spray dryer measuring station

air. The outlet air grasps the small fraction particles of the dried material with, but according to our tests this quantity means only a few per cent of the dried product.

During the measurements we controlled the quantity of the inlet solution by a butterfly valve on the suction side and the temperature by turning on and off the heating stages. In the experiments of spray drying we measured the important characteristics of the drying air:

- the temperature and wet thermometer temperature of the environmental air by the help of which the temperature of the entering air can be determined

- the temperature of the air entering the drying chamber
- the temperature directly behind the heat exchanger (for information)
- the temperature of the air leaving the dryer with the thermometer built into the bottom of the drying chamber
- the mass flow of the air by the measuring orifice built in before the fan, using a U–manometer.

The measuring points are shown on *Fig. 1*, too.

Spray Drying Investigations of Medicinal Plant Extracts

We carried out experiments to produce drug-extract in powder form from water containing extract of rose hip, camomile and lime blossom. The primary target of the investigations made was to find out if we could obtain good quality product in powder form by implementing this procedure.

We wanted to find out those technological characteristics of spray drying by which the procedure could be carried out so that the active principles suffer the minimum damage. Spray drying is a quite inflexible operation as the different technological characteristics have a close mutual influence on each other. The parameters cannot be changed in a wide range and independently from each other during the tests if we want to obtain a product that meets our requirements. This target got a priority for us during our experiments as we wanted to use the material obtained with our tests to work out further steps for the technology of producing pharmaceutical products.

We found out about all three extracts that the spray drying technology was suitable for drying these solutions.

We give you herewith a chart showing the most important characteristics of spray drying of medicinal herb extracts:

Table 1

Dried material:	T_1 °C	T_2 °C	m_1 kg/h	m_0 kg/h	m_p g/h	$\Delta h/\Delta Y$ kJ/kg	m_1/m_0
Rose hip extract:	153	65	259	5.09	364	7460	51
Camomile extract:	145	66	264	4.25	372	8390	62
Lime blossom extract:	150	64	266	4.67	150	7870	57

whereas the abbreviations have the meanings as follows:

T_1:	the temperature of the air entering the spray dryer
T_2:	the temperature of the air leaving the spray dryer
m_1:	the mass flow of the air used for drying
m_0:	the mass flow of the solution fed into the spray dryer
m_p:	the mass flow of the dry powder leaving the spray dryer
$\Delta h/\Delta Y$:	the specific drying air consumption for the inlet solution

From the data of the charts it can be seen that we carried out the drying under mild conditions in a way as it was our intention. We kept the temperature of the entering and leaving air low in order to avoid the heat sensitive components of the extract to be damaged. While the feed-in speed of the solution was low we used a great volume of air for the drying. This resulted in a relatively high air/solution specific rate.

It is quite clear that with the here described technological characteristics a very good quality medical plant extract powder can be produced but we can suppose that the product cannot be produced in a very economical way. To see if the drying technology can be intensified we made further experiments. These experiments were made on semi-industrial size devices to prepare the extracts and to do spray drying while changing the characteristic values of the drying in a wider range than before. These investigations were made to work out the industrial process for manufacturing these products.

The extraction was carried out in semi-industrial equipment. In some cases even additives were mixed to some solutions obtained by extraction according to the recipe of the Research Institute for Medicinal Plants. The solution prepared this way was brought into the spray drier. To determine the characteristic technological values we changed mainly the temperature of the entering and the leaving air during our investigations. We adjusted the proper solution input to these values while keeping the air mass flow at an approximately steady value. The quantity of the drug we had at our disposal set a limit to the number of the tests that could be made. When fixing the test points we gave a priority to the stationary and relatively steady running conditions. With these limitations we were trying to investigate the operating ranges that could come into consideration.

The device could be emptied and cleaned completely only after the individual test series and so the mass flow of the produced powder can be seen as an average value only.

We summed up the results of the test measurements in the following chart:

Table 2

Dried material:	T_1 °C	T_2 °C	m_1 kg/h	m_0 kg/h	m_p g/h	$\Delta h/\Delta Y$ kJ/kg	m_1/m_0
Rose hip 1	247	88	257	11.6	930	5530	22
	242	82	257	11.6	930	5420	22
	230	81	257	10.9	930	5470	24
	221	73	257	10.9	930	5230	24
	272	99	257	10.9	930	6560	24
Rose hip 2	260	80	257	16	1050	4240	16
	266	82	257	15	1050	4340	17
Camomile 1	261	100	257	11	650	6070	23
	239	84	257	11	650	5520	23
Camomile 2	266	85	268	13.9	550	5100	19
Lime blossom 1	255	100	260	10.8	—	6020	24
	232	85	260	10.6	—	5550	25
Lime blossom 2	239	80	274	11	300	5820	25
	234	75	274	13	300	4800	21

Evaluation of the Drying Results

It is to be seen from the chart that the spray drying of rose hip and camomile can be carried out without processing difficulties at an entering temperature of 240–260 and leaving temperature of 80–85 degrees centigrade. If necessary it can be tested by further investigations whether the individual active principles and aromatic components do not suffer damage due to the higher temperature applied.

We made the experience that the lime blossom extract is more heat-sensitive. It can be spray-dried, but very tenderly only. At the first series of tests the product got liquid due to the additives added and this is the reason why there is no leaving product shown in the summarising chart. At the second series of measurements the experts of Research Institute for Medicinal Plants used an additive that did not have a disadvantageous effect on how the extract could be dried. We made tests also with lime blossom extract without additive and found that it could be dried well.

It must be considered if it is necessary to mix additive to the extract before drying. May be it would be a better way to produce a higher concentration solution in a more step cascade and to spray dry this product.

In the charts we showed the specific energy consumption received based on the measurements. Analyzing them we can see that the heat energy 7500–8400 kg/h used up in the first series of measurements can be reduced to 4300–5000 kg/h if we intensify the drying method. This means a 35–40% decrease of specific energy consumption by choosing proper operating conditions while the productivity of the device grows to the double.

Summary

With our tests we could see that rose hip, camomile and lime blossom extract can be processed well in spray dryers. The powder obtained with this method serves as a raw material for further pharmaceutical products. Based on the investigation of the technology the most important characteristics of the industrial processing can be defined. The products can be produced safely with the determined values. Further on it is advisable to investigate the possibility of intensifying the extraction process in order not to have to use additives during the process.

References

KEDVESSY, GY.: Gyógyszertechnológia. Budapest, Medicina Könyvkiadó, 1981.

TOPÁR, J.: Mathematical Model of Spray Drying Reckoning with Droplet Size Distribution, *Drying 80*, Vol. 2. *Proceedings of the Second International Symposion*, Ed. A. S. Mujumdar, Hemisphere Publ. Co. Washington, 1980. p. 405.