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DEVELOPMENT OF NEW TYPE GAS HEATING DEVICE FOR NATURAL GAS PRESSURE REDUCTION STATIONS

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Abstract

The Petroleum Machine Works Budapest Corporation has developed a new construction gas heating device with cooperation of co-workers of the Chemical and Food Engineering Department at the Technical University of Budapest. The task of this device is: heating up the high pressure natural gas at the gas reception station before pressure reduction to avoid its cooling under the dew point.

Our department made measurements at the new heating device: examined its firing characteristics, its heat transfer characteristics, examined its control system circuits and the quality of its temperature control. We gathered operation experiences too about fulfilling the operational demands.

The examinations showed: the device working on the principles of closed two-phase thermosyphon satisfies the demands. The new injection type gas burner works well, it can be controlled well. The heat transfer in the heating device is good. The control system is satisfying the prescriptions.

Keywords: closed two-phase thermosyphon, gas heating device (boiler), heatpipe, pressure reduction station.

1. Introduction

The Petroleum Machine Works Budapest Corporation has developed a new construction gas heating boiler with cooperation of co-workers of the Chemical and Food Engineering Department at the Technical University of Budapest for heating up high pressure natural gas at gas reception stations.

The Petroleum Machine Works Budapest Corporation built up the gas heating boiler on the basis of preliminary calculations and set to work at one of his working sites. At this device the co-workers of Petroleum Machine Works Budapest Corporation and our department made examinations concerning of its operation characteristics and its optimal operational settings.

2. Task and Construction of Natural Gas Receiving Stations

The main task of gas receiving stations is the pressure reduction of high pressure gas — 6.4, 3.2, 1.6 MPa — to the user-needed low: 1.2, 0.6, 0.3 MPa pressure. The simplified flow sheet of the gas receiving station can be seen at *Fig. 1*.

The regulated pressure reduction is realized by expansion valve (GSZ). During the pressure reduction the temperature of the gas decreases and at low temperature the high viscosity paraffin compounds and hydrates are condensing out of the gas and these substances may block the valve. In order to prevent this operational problem the primary: high pressure gas must be heated up to a necessary extent. The natural gas is heated in (FK) boiler with gas burner to which the necessary gas is taken from the secondary low pressure side. The receiving station is provided also with measuring, controlling and safety elements furthermore fittings.

The receiving stations are mostly built up outside of inhabited areas, therefore they are remote controlled ? behalf of telemetric lines. The gas receiving stations have very low electric energy consumption. In case of the electric supply breakdown they run from batteries for a given limited time. In case of breakdown of the burner the primary gas heating must be provided until maintenance staff arrives.

The task of the natural gas preheating boiler is to elevate the temperature — the enthalpy — of the primary (high pressure) gas to such an extent, that the decreasing temperature of the expanding gas won't reach dew point temperature: 3 °C.

At the used primary (high) and used secondary (low) pressures the temperature decrees of the natural gas can be characterized by about $\Delta T/\Delta p = 0.4$ K/bar [1]. When gas consumption, composition of the gas and the temperatures are known, the heat consumption can be determined, the preheating boiler can be designed, or for a given receiving station it can be chosen from series of different capacity boilers.

3. Old Type Natural Gas Preheating Boilers

The old type natural gas preheating boilers have big heated water reservoirs. The heat exchanger tubes through which the primary gas flows are immersed into the hot water. The heating of the high pressure gas is realized from outside by free convection of the hot water. The heating of the heat transmitting fluid (water) is maintained by a gas burner. For temperature control the burner can work on full load, on a 43% load and can be switched off. The built in heating capacity must satisfy the maximal needs.

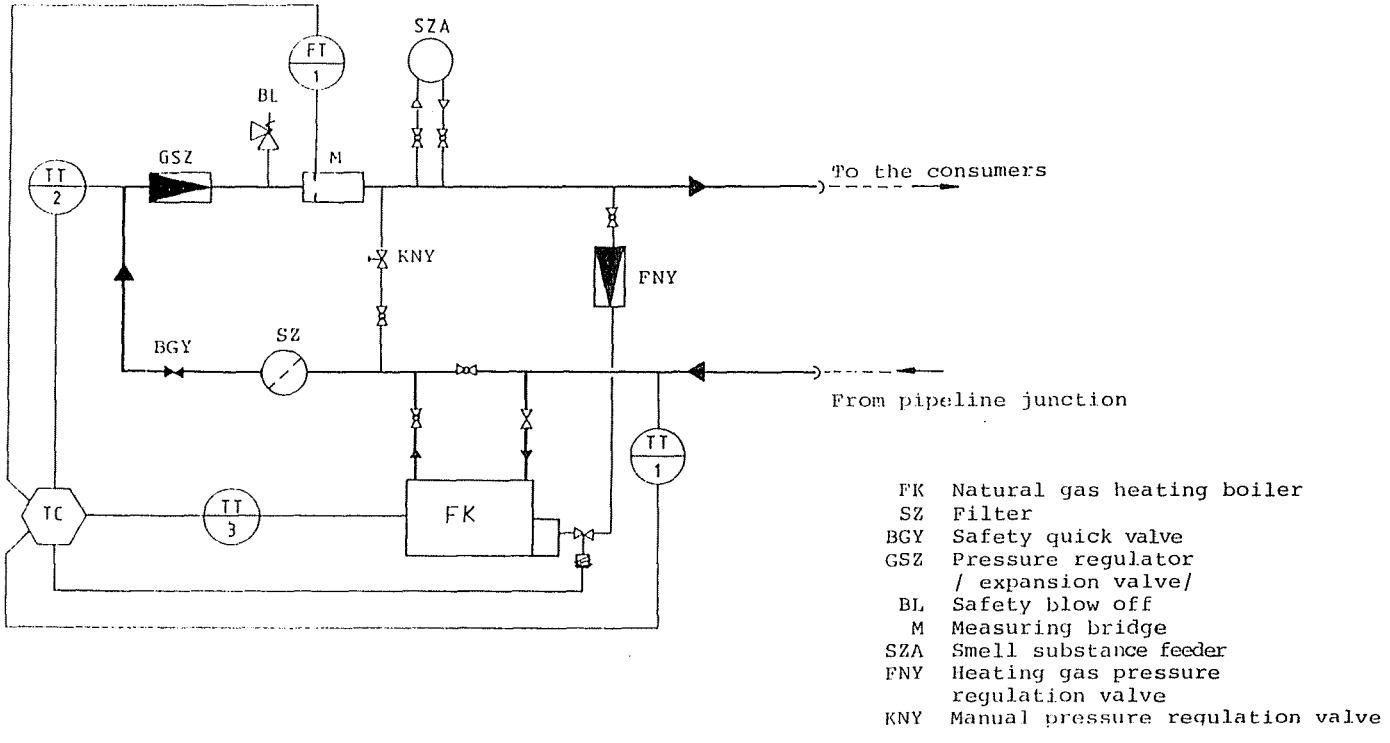

Fig. 1. Sketch of gas receiving station

The described regulation is too rough and leads often to overheating: to waste of energy.

The old type boilers which were installed long ago are obsolete and physically worn-out. They must be exchanged for new ones. They are obsolete because they don't meet the environmental needs and energy saving prescriptions. This was the reason which led to the development of the new type.

4. The New Type Natural Gas Preheating Boiler

The main directives to the construction of the new type's development were: good regulability, dynamic response on varying needs of the consumers, satisfaction of prescriptions on environmental protection and safety.

The old type was built in two sizes: with 93 and 325 kW output. The economical operational range of the two sizes was far from each other. When they were operating at loads between 43% and 100% the control system switched between these two states. At loads under 43% — this is equal to 40 kW in case of the smaller and 140 kW in case of the greater size — the control system couldn't work normally: it overheated the water and then switched the burner off. It operated by switching the burner on and off.

At the new type the intention was to make a series of sizes to meet the needs of the consumers between great load differences. It seemed: sizes of 100, 200, and 400 kW output will realize this intention. On the other hand, it is necessary that their economical operation ranges should overlap each other.

The task of the gas heater is to hold the temperature of the gas after expansion on an expected value or in a required narrow temperature interval — even if the mass stream, pressure and temperature of the gas change. The simplified flow sheet of the new gas heater is shown on *Fig. 2*.

The new boiler works on principles of closed two-phase thermosyphon. It consists of a cylindrical — pressure and vacuum tight — vessel, which is filled by working fluid 6 — water — to an appropriate height. The flue pipes and the burner space — 3 — is surrounded by the water. On their surfaces the water is boiling. The produced steam is gathered in the steam space — 7 —. The steam condensing on the cold surface of the high pressure (primary gas) heat exchanger — 8 — gives his phase-change heat to the gas and the condensed water drops back to the water space.

In the water space of the boiler there is a heat transfer characterized by boiling and in the steam space there is condensation — both realize a very good heat transfer coefficient. This is the main difference to the

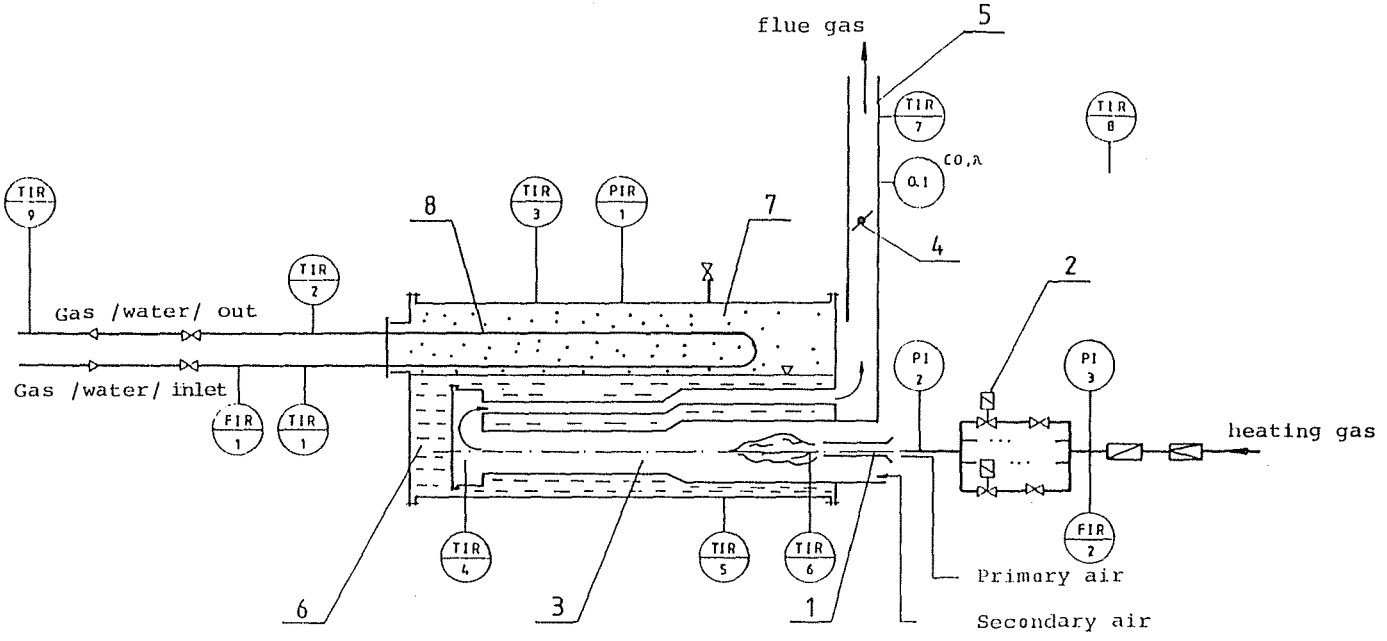

Fig. 2. Theoretical sketch of gas heating boiler and its instrumentation

1. Gas burner
2. Solenoid valves
3. Fire tube
4. Air clack
5. Chimney
6. Water space
7. Steam space
8. Gas heating heat exchanger

old type, where the heat transfer was realized on the heat exchanger tubes immersed into the water by free convection — characterized by poor heat transfer coefficient. Therefore the heat transfer in the new type can be realized on much smaller surface. Another advantage of this new system is that the good heat transfer makes the regulation better.

5. Examination of the New Gas Heater

On the new gas heater the following examinations and measurements had to be made:

- firing examinations, concerning the examination of burning of the gas and composition of the flue gas;
- heat transfer measurements;
- examination of the temperature control system and its adjustment;
- operational experiences and comparing them with the expectations.

The measuring arrangement had to be shaped so that it met these demands.

The prototype had been provided with temperature, pressure and mass flow transmitters which made quick measurement of several parameters, data acquisition, and analysis possible.

During the examination the following analog signs were measured:

– temperature of the steam space	$T_{c'}$	TIR 3
– pressure of the steam in the boiler	$p_{c'}$	PIR 3
– volume flow of heating gas	$V_{G'}$	FIR 2
– burning temperature	T_{burn}	TIR 6
– wall temperature of the burning space	$T_{W1'}$	TIR 5
– wall temperature at return point of burning space	$T_{W2'}$	TIR 4
– flue gas temperature in the chimney	T_{flue}	TIR 7
– ambient temperature	$T_{amb'}$	TIR 8
– volume flow of cooling water	$V_{V'}$	FIR 1
– inlet temperature of cooling water	$T_{in'}$	TIR 1
– outlet temperature of cooling water	T_{out1}	TIR 2
– outlet temperature of cooling water far from boiler	T_{out2}	TIR 9

The quick registration and processing of this great number of data we solved by using PC-AT based on line data acquisition system.

With the data acquisition system we collected two level signs as well. Most interesting of them were those which showed the number of solenoid valves supplying gas to the burner. (This number gave information on the number of open and closed solenoid valves). We registered the status of the

safety limiters, too, the status of which had to be taken in consideration at the control of the burner.

Data which weren't switched to data acquisition and were measured time to time with conventional method were:

– pressure of inlet gas	$p_{iG'}$	PI 3
– pressure of gas before the nozzle	$p_{n'}$	PI 2
– measurement of smoke composition	x, λ	QI

5.1 Firing Examinations

Flow rate and pressure of the gas to the injection type burner 1 at *Fig. 2* can be remote switched and can be adjusted by valves 2 for the needed heat performance. The gas burns in burner 1 mixed with primary and secondary air stream and gives his caloricity to the flue gas. The flue gas streams through the fire tube 3, the return band and flue tubes and leaves the boiler through the chimney 5 to the open air after it has given down its heat content. The flue gas stream can be controlled by clack-valve 4 in the chimney.

There is a difference between the new and the old type in volume of the water space, too: the old type contains 7 or 14 m^3 — the new one only 1.8 m^3 water. The smaller water space has smaller heat inertness therefore it can be controlled easier. On the other hand: its heat capacity is great enough to heat up the primary gas until the fitters can arrive and repair a breakdown of the burner.

The new type boiler can be controlled in more steps than the old one. The heating gas can be led with help of solenoid valves between 40% and 100% nominal load in 4 steps to the burner and adjusted this way to the heat demand. The burner which was developed by TÜKI to this new type boiler can be modified by changing the nozzle and by settings of the air traps to 100, 200 and 400 kW nominal output.

The new type boiler can be controlled in a greater range and in more steps than the old one. Smaller water space and better heat transfer make the dynamic characteristic of the new type better.

Regulation is realized by a newly designed electronic device and this fulfills the realization of blocking for safety prescriptions as well.

Firing technical examinations had not only the setting of the burner in view but the measurement of the coming out flue gas as well, being important for fulfilling environmental and health regulations.

We determined the following parameters with a flue concentration measuring instrument:

T_{fg}	°C	temperature
x_{O_2}	v/v%	oxygen content
x_{CO_2}	v/v%	carbon dioxide content
x_{CO}	v/v%	carbon monoxide content
x_{SO_2}	v/v%	sulphur dioxide content
x_{NO_x}	v/v%	nitrogen oxide content
λ	–	excess air coefficient
η	%	firing efficiency

These measurements made possible to adjust the firing technical characteristics to various nominal heat outputs.

5.2 Thermal Measurements

In the gas heating boiler heat transfer is realized in two steps.

The thermal energy from the burning gas is transferred on the surface of the flue space from the flue gas to the water, when at the whole surface — in consequence of overheating — boiling heat transfer and steam production is realized. At this process the flue side heat transfer coefficient is the governing one.

In the steam space over the water space the condensation heat of the steam is transferred to the high pressure gas streaming in the heat exchanger tubes. Here the gas side heat transfer is the governing one and this will regulate the measure of the heat transfer coefficient. We show simplified the whole heat transfer and the characteristic temperature profile in the gas heating boiler on *Fig. 3*. (This figure doesn't show that the greater part of the heat amount is transferred in the fire tube by radiation.)

The broken lines of T_{evap} and T_c are in the reality nearly equal. Their value changes in function of thermal load.

In the case of the thermal measurements we examined basically the steady-state conditions of the gas heating boiler, at conditions the transmitted heat of the flue space is equal to the accepted heat of the gas.

In the realization of the measurements the trouble was, that the gas to be heated has 64 bar pressure. On the basis of the measurement plan for the prototype examination the volume stream of the gas should have been changed and the heated gas should have been used up. In order to solve these technical, safety and economical problems, we made the measurements with water as cold medium and using the rules of similarity we deduced to working circumstances the probable heat transfer results. The argument for using water as cold medium was the fact that physical

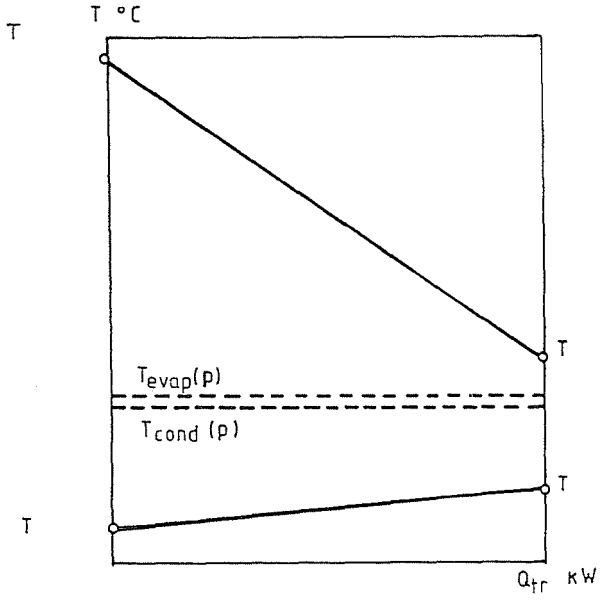

Fig. 3. Approaching temperatures in function of heat

properties of water were nearer to 64 bar natural gas as those of neutral near atmospheric pressure gases (e.g. air, nitrogen) would have been.

Results of some measurements can be seen in *Table 1*.

In the boiler a two step heat transfer is realized: the flue gas heats the water and the generated steam heats the high pressure gas — during our measurements the cooling water — in the form of condensing on the heat exchanger tubes in the steam space. The heat transfer between the burner space and the water space is independent of the cooling medium. If the temperatures are same during the measurements and at working circumstances the heat transfer coefficients will be the same here as well. The second heat transfer step is: heat transmission from the condensing steam to the streaming cold medium. Here the smaller heat transfer coefficient is at the streaming medium side and this will govern the heat transfer. Analyzing the results of the measurements it turns out that the stream is turbulent.

In measurements shown we determined the Reynolds numbers of the cold medium — water — and from the measured α_W heat transfer coefficients the values of measured Nu numbers. For comparison we determined the Nu number by the help of the following Gnielinski equation being valid

Table 1
Results of some measurements of gas heating boiler

Sign of meas.	$Q_{\text{trans-mitted}}$	Access air coeff.	Firing eff.	T_{fg}	p_C	T_{in} (water)	T_{out1} (water)
	kW	λ	$\eta\%$	°C	bar	°C	°C
1.	142.2	1.28	94.4	125.0	2.0	10.0	32.7
2.	–	2.66	90.0	115.5	1.52	10.1	37.4
3.	173.5	1.18	94.6	132.0	1.91	10.6	36.5
4.	112.0	1.7	91.0	117.0	1.7	7.1	34.2

Sign of meas.	$\dot{m}_W$ (water)	$k_{\text{fire tube}}$	$k_{\text{flue tube}}$	k_W	α_W	x_{CO_2}	x_{CO_2}
	kg/s	$\frac{W}{m^2K}$	$\frac{W}{m^2K}$	$\frac{W}{m^2K}$	$\frac{W}{m^2K}$	v/v%	v/v%
1.	1.50	42.5	23.3	711.6	848	0.025	9.3
2.	0.77	21.2	23.7	490.0	551	n.d.	4.5
3.	1.60	51.4	22.6	912.4	1184	–	10.1
4.	1.11	38.1	23.4	672.0	791	n.d.	n.m.

in turbulent stream. (The equation can be found in VDI Wärmeatlas [2].)

$$Nu = \frac{\xi/8(Re - 1000)Pr}{1 + 12.7\sqrt{\xi/8}(Pr^{2/3} - 1)} \left[1 + \left(\frac{d_G}{l}\right)^{2/3}\right],$$

where

$$\xi = (1.82 \log_{10} Re/1.64)^{-2}.$$

Table 2
Comparison of measured and calculated Nu numbers

Sign of meas.	Re	Nu measured	Nu calc.	Error %
1.	12433	66.8	84.59	21
2.	6382	43.4	45.7	5
3.	13262	90.4	89.58	1
4.	9201	62.3	64.48	3.4

We found differences of 1 to 21% between measured and calculated Nu numbers at same Re numbers. On the basis of this result it is clear:

the Gnielinski criteria equation describes the heat transfer well and it can be used for determination of heat transfer coefficient of the streaming high pressure gas at working circumstances.

The main difference between heating up high pressure gas and heating up water is caused by the difference of the specific heats:

$$C_W = 4.187 \text{ kJ/kg K} \quad \text{and} \quad C_{pG} = 2.765 \text{ kJ/kg K}.$$

This results: 33.4% higher mass stream gas can be heated up at a same temperature difference as water could be. Difference is caused also by the Prandtl-number difference:

$$Pr_W = 4 \quad \text{and} \quad Pr_G = 0.873.$$

For comparison of high pressure gas heating and measurements with water we made the following calculations. If we heat up 64 bar high pressure gas similarly the shown 3. measurement with 173.5 kW and with the needed $\Delta T = 30$ °C temperature difference, the mass stream of the gas will be:

$$\dot{m}_G = 2.092 \text{ kg/s} = 7530 \text{ kg/h}.$$

The velocity of the gas in the tube is

$$u_G = 7.79 \text{ m/s}.$$

In this case the Re number:

$$Re = \frac{u_G d p_G}{\mu_G} = 1.445.000.$$

Calculating with $Pr = 0.873$ and Gnielinski equation [2]

$$Nu = 1948.8.$$

The heat transfer coefficient from this:

$$\alpha_G = 1446.3 \frac{W}{m^2 K}.$$

Taking the heat resistance of the condense film and tube wall in consideration we get an overall heat transfer coefficient:

$$k = 1090 \frac{W}{m^2 K}.$$

The measured device has 2.026 m^2 heat transfer surface in the steam space, the driving force of heat is:

$$\Delta T_{lg} = 78.5 \text{ °C}.$$

If 5 °C is the incoming temperature there must be $T_C = 99.4$ °C and $p_C = 0.999$ bar pressure in the steam space in order to maintain the $\Delta T = 78.5$ °C.

5.3 The Structure and the Examination of the Temperature Control System

The purpose of the temperature control shown in *Fig. 1* is to ensure the natural gas quantity needed for the natural gas not to cool down under the dew point during the expansion. *Eq.* (1) consisting of the temperature and the pressure before and after the expansion valve is used to determine the natural gas temperature before the expansion valve, which must be the set point of the temperature controller

$$T_a = 0.4(p_{pr} - p_s) + T_s. \tag{1}$$

The temperature T_s after the valve is determined together by the dew point and the temperature controlling uncertainty. As the dew point of the natural gas is -3 °C and the maximal allowable error for the controller is 3 °C, thus $T_s = 0$ °C. *Table 3* shows the typical temperature set point values at the usual primary and secondary pressures.

Table 3
Typical temperature basic signs

p_{pr} bar	p_s bar	T_a °C
64	12	20.8
	6	23.2
45	12	13.2
	6	15.6
22	12	4.0
	6	6.4

In the structure of the temperature control system the steam space temperature (T_C) of the boiler has a prominent role. T_C can be calculated from the equality of the heat transferred on the surface (A) of the gas heater and heat taken up by the high pressure gas at steady-state conditions.

$$\dot{Q}_G = \dot{Q}_C, \tag{2}$$

$$\dot{Q}_G = \dot{m}_G c_{pG}(T_{Gout} - T_{Gin}, \tag{3}$$

$$Q_k = kA\Delta T_{mean}, \tag{4}$$

$$\Delta T \sim T_C - 0.5(T_1 + T_2), \tag{5}$$

$$T_C = T_{Gout}(c_1\dot{m}_G + c_2) - T_{Gin}(c_1\dot{m}_G - c_2), \tag{6}$$

$$c_1 = \frac{c_{pg}}{kA} \quad \text{and} \quad c_2 = 0.5. \tag{7}$$

To produce the heat quantity defined by *Eq.* (2) under steady-state conditions and neglecting the losses we have to burn as much natural gas as *Eq.* (8) says.

$$\dot{m}_t = \frac{Q_C}{\eta H_a}. \tag{8}$$

Eq. (8) gives the functional relationship between T_C and $\dot{m}_t$.

$$T_C = \frac{\eta H_a}{kA}\dot{m}_t + 0.5(T_{Gin} + T_{Gout}). \tag{9}$$

Fig. 4 shows the inner structure of the (TC) symbolized temperature controller in *Fig. 1*.

Fig. 4 shows that four discrete gas rates can be switched with the on/off temperature control structure. Experiments were made using the prototype with control structure shown in *Fig. 4*, using water instead of natural gas. This means that the control system was a little bit different from the original one designed for the real circumstances. Despite that fact we were able to do the examination of the response curves for the process and the examination of the controller during working. The responses of the system to unit step inputs are shown in *Figs 5* and *6*. The cooling water mass flow was kept on 0.35 kg/s, the input temperature was 17 °C so it was kept on constant value during the 7 measuring hours. The set point of the controller was 28 °C.

The useful heat quantity needed to warm up the natural gas was maximally 180 kW produced by a heating gas rate of 24 Nm^3/h at steady-state conditions. *Fig. 5* shows that the temperature control was stable. During our experiments it was proved that the control system is suitable for steady-state operation. *Fig. 6* shows the change of the manipulated variable in time. It shows a cyclical operation. If 4 solenoid valves are open, the cycle time is equal to 113 minutes where the warming up period is 22 minutes long and the cooling back time lasts 91 minutes as clearly shown by the figure. If 2 solenoid valves are working the heating up takes 13 minutes and the cooling down 48 with a whole cycle time of 61 minutes.

To determine the peak-to-peak amplitude, the overshoot, and the rise time, we have to examine the response of the system between 110 to 140 and 390 to 420 minutes on *Figs 5* and *6*. It can be seen that there is a change in the temperature of the flue gas $\Delta T_{(flue)} = 101.5 - 69 = 32.5$ °C and in the temperature of the steam space $\Delta T = 87 - 70 = 17$ °C relative to the work point data. The dead time is less than 1 minute in the flue. After switching out the heating, the burning space cools down almost

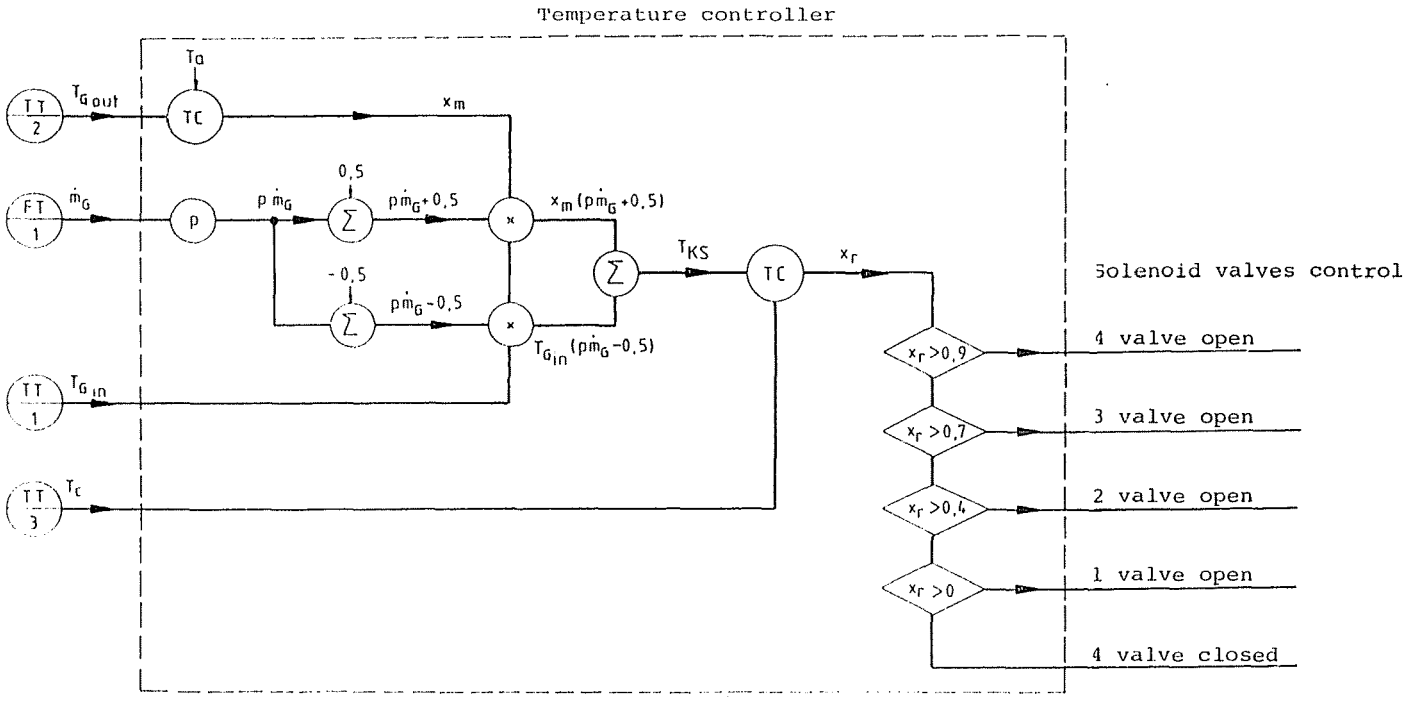

Fig. 4. The structure of temperature control system

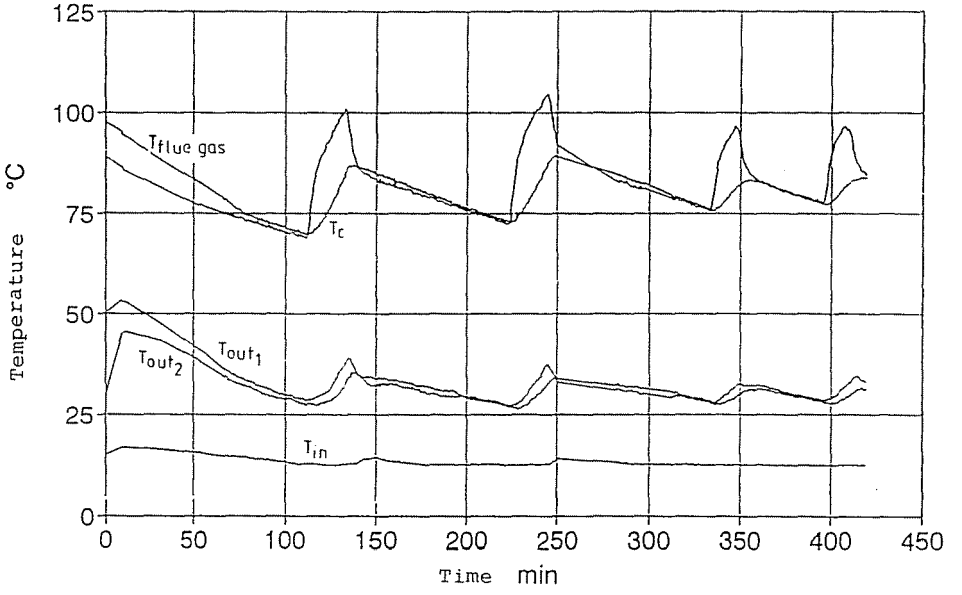

Fig. 5. Plotted results of temperature controller

immediately as we can see in the figure clearly. The overshooting of the boiler's steam space temperature is less than 1 °C. The directly controlled system has an effective dead time of 8 minutes. The temperature rise in the steam space using 24 Nm^3/h burning gas rate is equal to:

$$\frac{\Delta T_C}{\Delta t} = \frac{17\ ^\circ C}{14 min} = 1.2 \frac{^\circ C}{min}.$$

We can see that the temperature change caused by the intervention at the outlet of the cooling water is equal $\Delta T = 38.7 - 28.5 = 10.2$ °C and at the temperature of the distant cooling water is $\Delta T = 35.5 - 27.5 = 8$ °C. From the figure we can see that in that moment when we switched out the heating the temperature was $T_{c2} = 33$ °C, but because of the 4 minutes dead time the system warmed up to 35.5 °C. It is ascertainable that until 10 minutes there isn't any significant rise of T_{c2} temperature. After the dead time the temperature rise was:

$$\frac{\Delta T_{c2}}{\Delta T} = \frac{8\ ^\circ C}{12\ min} = 0.7 \frac{^\circ C}{min}.$$

It appears from the figure that the controlling band gets narrow if we burn 19.3 Nm^3/h gas for a shorter 11.2 minutes time. Finally we can say that

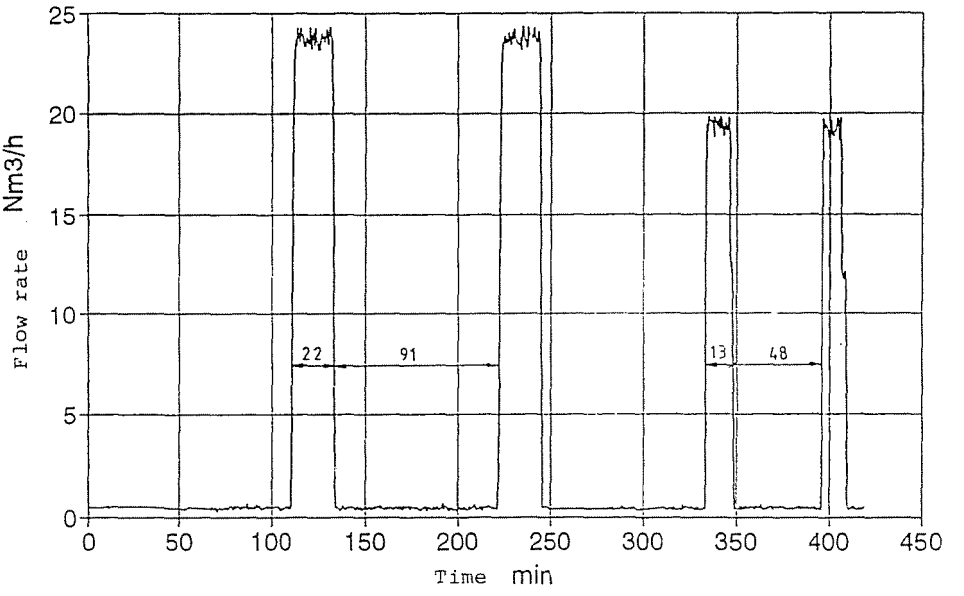

Fig. 6. Manipulated variable

the steady-state deviation is not constant. The steady-state deviation in the first case was 7.5 °C and −0.5 °C, and in the second case 3.5 °C and −0.5 °C.

6. Summary

The new type gas heater developed and manufactured by Petroleum Machine Works Budapest Corporation is built for 3 nominal capacities. The prototype had been examined by the Chemical and Food Engineering Department of the Technical University of Budapest.

During the measurements its units and functions were examined separately, too. The examined capacity settings were in the probable working capacity ranges.

The injection type gas burner worked normally and safe.

The nominal 100, 200 and 400 kW capacity can be provided by changing the nozzles and by regulation of the primary air. The secondary air

streams free through orifices around the burner in accordance of actual draught.

This causes too much excess air at small outputs, but this could be installed to the normal firing technical values in function of the gas load by the air clap in the chimney. On the basis of our working experiences we recommend the modification of the secondary air inlet.

Our opinion is: it would be useful to provide the final design of the gas heater with continuously working gas feeding for the burner — instead of the present stepwise regulation of heating and promising gas supply of the burner in continuous accordance of heat demand. This would improve regulation stability, too.

It would be useful to mount in regulator for secondary air supply: this could use the air clap in the chimney for controlling. This regulation could hold the firing efficiency on nearly optimal value.

The measured prototype was destined originally in order to represent the three different capacity boilers 'built together'. During the examinations we got operational experiences and problems which must be taken into consideration in the final design.

The measurements on the first variant of the prototype boiler showed clearly: the construction of the flue tube must be altered because every heat capacity step needs another flue tube and another return band construction. The measured first variant cooled the smoke at small capacity to a too low temperature and its pressure drop was too high: there were draught problems at 300 kW capacity. After the experiences of the first measurements the Petroleum Machine Works Budapest Corporation modified the equipment: they built a new flue tube which was fitted to the 100 kW capacity. We finished our examinations on this modified equipment. There were no draught problems and we got proper flue gas temperatures: over the required 120–140 °C. In both operational and safety respects it worked properly.

From viewpoint of control it would be useful to make the water content and the mass of the boiler smaller for decreasing the dead time. But this is limited by the fact that in case of breakdown of the heating the accumulated heat must warm up the gas in the time interval of breakdown and starting the burner by the technician.

The examination of the control system showed: the controlled system has dead time and integrating characteristic in the neighbour the work point. Cascade control structure built up from PI character parts can hold the set point with 3 to 10 °C error constant. It is advantageous: we found the high steady state deviation with $\sim +3$ to 8 °C over the set value. The control can follow well the slow ($\sim$ 1 °C/min) work point alterations — hood are caused by the disturbances —. The controller and the controlled

system must be fitted in spot where the whole equipment is installed taking in consideration the real circumstances .

Because of the high steady-state deviation we recommend to use continuously working control systems instead of the built on-off control structure.

Notations

A	heat transfer surface	t	time
c, c_p	specific heat	T	temperature
d	distance, diameter	ΔT	temperature difference
H_a	caloricity	u	velocity of medium
k	overall heat transfer coefficient	V	volume stream
$\dot{m}$	mass stream	x	concentration
Nu	Nusselt number	α	heat transfer coefficient
p	pressure	η	firing efficiency
Pr	Prandtl number	λ	excess air coefficient
$\dot{Q}$	heat flux	μ	dynamical viscosity
Re	Reynolds number	ρ	density

Subscripts

a	set point	C, $cond$	condensation, steam side
amb	ambient	$mean$	mean
in	inlet	tr	transported
$burn$	firing, burning	lg	logarithmic
$w1$, $w2$	wall	pr	primary
$flue$	flue gas	s	secondary
$evap$	evaporation	t	gas supply
G	gas	w	water
$out1$, $out2$	outlet		

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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

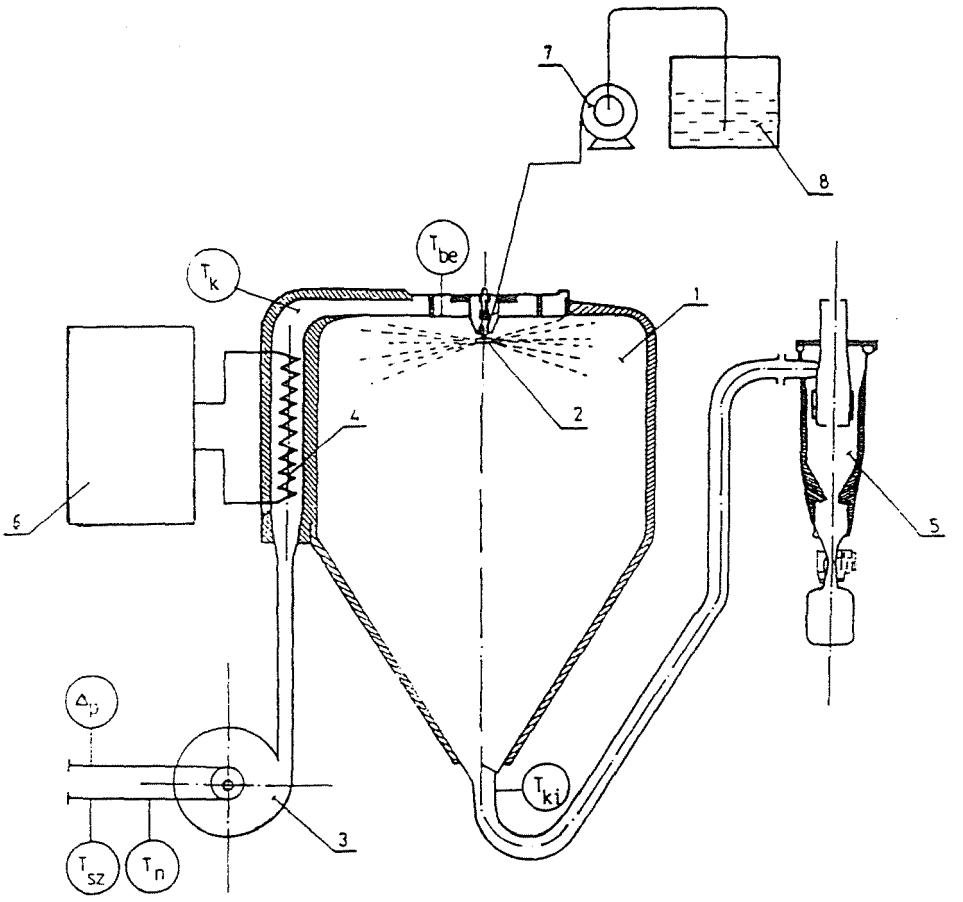

Fig. 1. Spray dryer measuring station

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.

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