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RESEARCH PAPER

Single-Step Granulation: Development of a Vacuum-Based IR Drying Method (Pilot Scale Results)

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ABSTRACT

A simple placebo granulation was dried either by an exclusive vacuum method or by combined microwave, strip-gas, infrared, and vacuum methods in a 50-liter pilot scale single-step granulator (Zanchetta Roto P). The results were compared.

INTRODUCTION

The purpose of this study was to develop an improved granule drying method which combines surface and core drying components, and is based on the very common and well-understood drying mechanisms vacuum, conduction, convection, and infrared (IR) drying. It should be tested whether a combination of the above-mentioned drying components could be equivalent or perhaps superior to a microwave-based drying process published previously (1,2) in terms of drying times, and whether it is able to meet the criteria of a universally applicable production method.

MATERIAL AND METHODS

The pilot scale drying experiments were carried out in a Zanchetta Roto P 50 single-step granulator (Zanchetta & C.s.r.l., I-55010 Lunata) with the same placebo formulation as described in a previous paper (2). The batch size was 12.0 kg. In order to standardize the granulation process all parameters were kept identical within a very narrow range in order to have a good reproducibility. The granulation process was subdivided into the three steps, each of them lasting 3 min:

I. Dry mixing: impeller 150 rpm; chopper 1500 rpm

II. Binder addition: impeller 150 rpm; chopper 1500 rpm
III. Wet mixing: impeller 280 rpm

Chopper action was only necessary in order to improve mixing in the first phase and binder distribution in the second. During agglomeration, impeller action at high speed produced stable granules without lumps.

The binder liquid was added through a 2-mm spraying nozzle at a pressure of 2.5 bar and had a temperature of 57.0 ± 1.0°C. The addition took 80 ± 5 sec and for another 100 ± 5 sec the impeller and the chopper speed were kept at the same level until phase III started.

During the drying step the pressure inside the vessel and the temperature of the material were registered. The moisture content of the batch was determined by a Mettler IR balance LP 16 at 105°C (Mettler GmbH, D-35396 Giessen) (3). The determination was stopped automatically when the loss on drying (LOD) was constant over a period of 3 min.

During drying the whole mixer was continuously tilting at an angle of 120°. Additionally intermittent mixing of the impeller was programmed with a defined on/off cycle (6 sec/54 sec). This way the formation of large agglomerates could be avoided and sufficient heat transfer from the wall into the batch could be obtained.

Two different kinds of IR equipment were used: a ceramic radiator Elstein T-FSR 1000 (Elstein-Werk M. Steinmetz AG, D-37154 Northeim) with a maximal emission wavelength of 3.0 μm and a quartz twin-tube radiator (Heraeus Kurzwelle) with an emission maximum of 1.3 μm (Heraeus Quarzglas GmbH, D-63801 Kleinostheim). Two possible types of quartz glass were tested in preliminary trials: quartz glass HOQ 310 and quartz glass Infrasil 302 (Heraeus Quarzglas GmbH, D-63454 Hanau). Both were suitable as vacuum-tight IR windows because of their excellent mechanical stability and their low content of hydroxyl groups (<30 ppm), but the cost–benefit relation was better for HOQ 310. Due to the low hydroxyl content, which is achieved through a special production procedure, only small amounts of IR radiation are absorbed by the quartz glass window itself.

Heating of the strip-gas was done by a Leister fan (Leister, CH-6056 Kaegiswil). Strip-gas temperature was controlled by a PT 100.

RESULTS AND DISCUSSION

Combined Vacuum and Contact Drying

In order to have a reference which was to be compared with other results, some trials were carried out with a combination of the two drying mechanisms: vacuum and contact drying. By varying the jacket temperature and the vacuum level, significant differences concerning drying time could be observed (Fig. 1). (*Note*. In the figures indicating drying results, the drying parameters are listed after the trial number: e.g., E 260322. Dotted lines indicate temperature curves.)

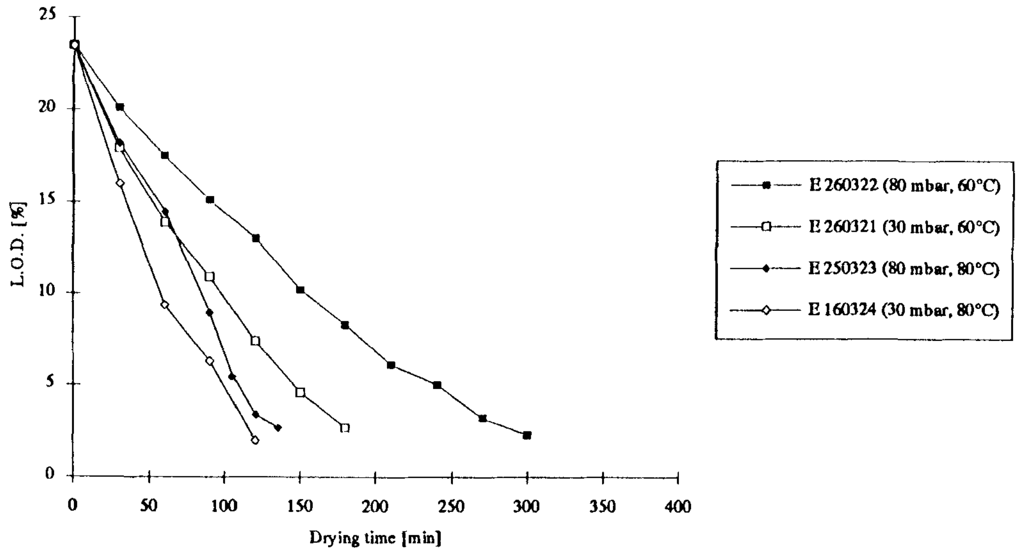

Figure 1. Drying rates at two different drying temperatures and pressure levels.

The explanation for these phenomena is given by the temperature of the jacket, on the one hand, and the boiling point of water at the pressure inside the vessel, on the other hand. The resulting differences between these temperatures are in good correlation with the drying times observed (Table 1).

Combined Vacuum, Contact, and Strip-Gas Drying

As preliminary trials had proven a dependency of the drying results from environmental conditions (4), especially humidity and temperature of the air, the drying conditions had to be standardized with respect to these factors. Instead of unconditioned air, compressed air with a residual moisture content of 0.7 g/kg dry air was used; its flow was measured by a flow meter and its temperature was modulated by a heating fan and controlled by a PT 100 temperature measurement system (Fig. 2). The input of the strip-gas into the granulated batch was accomplished by porous sintered metal inserts in the back of the three impeller blades.

Although the calorimetric capacity of gases is rather low, a significant increase in drying rate could be observed in one trial (E 141021) by moderately heating up the strip-gas compared to a trial (E 271024) where the strip-gas was not heated at all (Fig. 3).

As the corresponding boiling temperatures of water—57.0°C at 185 mbar (E 141021) and 58.3°C at 173 mbar (E 271024)—were quite similar, the different runs of the drying curves were mainly due to the temperature of the strip-gas. From the temperature of the batches at the end of the drying process (E 141021: 46.6°C; E 271024: 49.2°C) another conclusion could be drawn: The increase of the strip-gas temperature was increasing the moisture uptake of the air rather than warming up the granules.

An improvement of the drying rate could be observed up to a maximal flow rate of 17,150 liters/hr, which was indicated by lower residual moisture values in the corresponding batches after a drying time of 105 min (Fig. 4). Above the optimal flow rate the drying results became worse while the pressure inside the vessel increased continuously. For every dryer and vacuum pump combination the optimal volume flow rate has to be determined empirically.

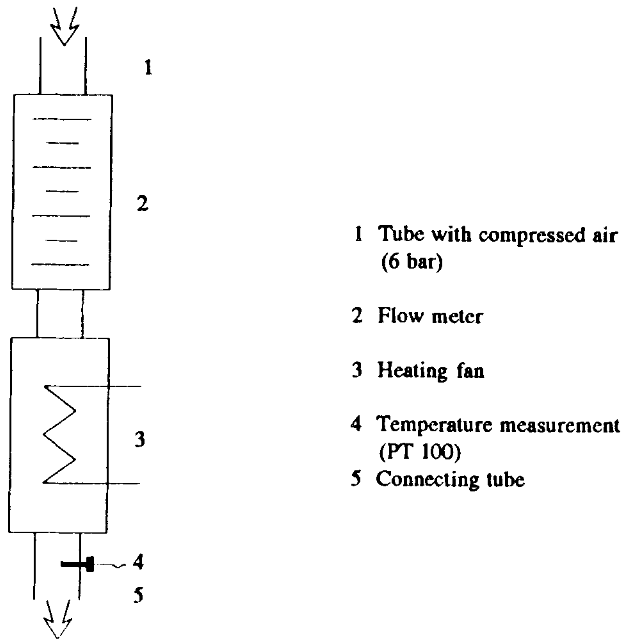

Figure 2. Device for a flow- and temperature-controlled air supply.

Combined Vacuum, Contact, Strip-Gas, and IR Drying

In order to reduce drying times even further, the usability of different IR radiators should be tested for this purpose. As preliminary drying tests with a static bed had shown that the penetration depth of IR radiation was in the range of approximately 1–2 mm (4,5),

Table 1

Trial	Temperature 1 (Jacket) (°C)	Pressure (mbar)	Temperature 2 (Boiling Point of Water) (°C)	Difference T1 - T2 (°C)
E 260321	60	30	24	36
E 260322	60	80	42	18
E 160324	80	30	24	56
E 250323	80	80	42	38

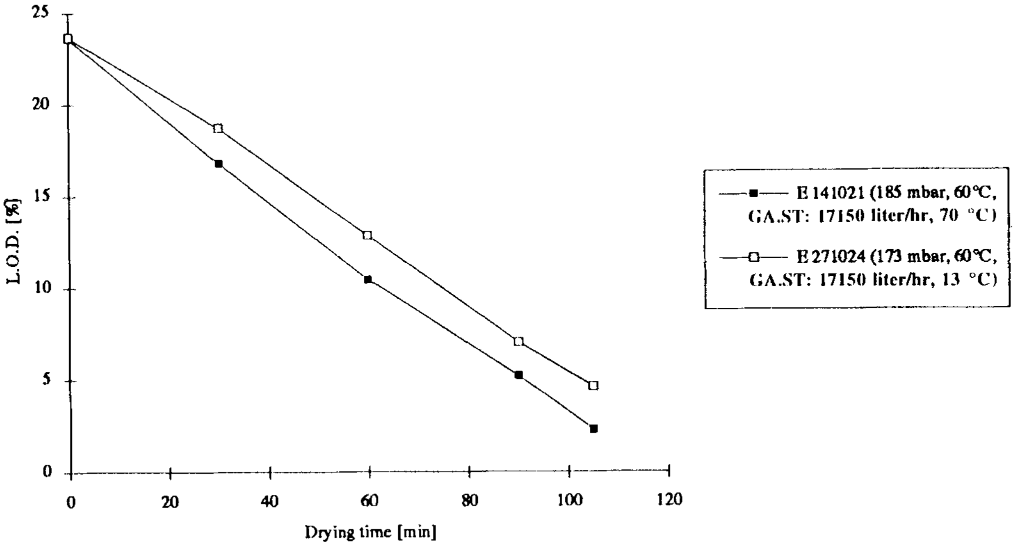

Figure 3. Drying rates with additional strip-gas of different temperature levels.

an effective mixing of the batch surface had to be guaranteed in order to distribute the absorbed radiation energy.

At the same time some additional preconditions had to be fulfilled. Mixing by the impeller was not a suitable way to achieve effective and gentle mixing of the surface granules—because the permanent impeller action would result in total destruction of the granules. The tilting option of the whole mixer turned out to be a satisfactory solution of the problem, thus avoiding high attrition the surface of the batch, which was constantly changing by an essentially passive mode of mixing. As only the center of the batch surface was irradiated and as the impeller was completely covered by granules, there were no static conditions with the risk of local overheating or burning.

The IR radiators could not be installed directly above the granule surface because of their high working temperatures of about 700°C and about 800°C, respectively (Fig. 5) (6). Therefore the energy had to be transferred through a window (3) in the mixer lid. Due to their mechanical, and especially their optical, properties, only certain types of quartz glass were suitable because their transmission in the IR range above 3 μm wavelength was about 90%.

Nevertheless the quartz glass heated up by absorption of the residual 10% of radiation so that it had to be cooled by a fan (5).

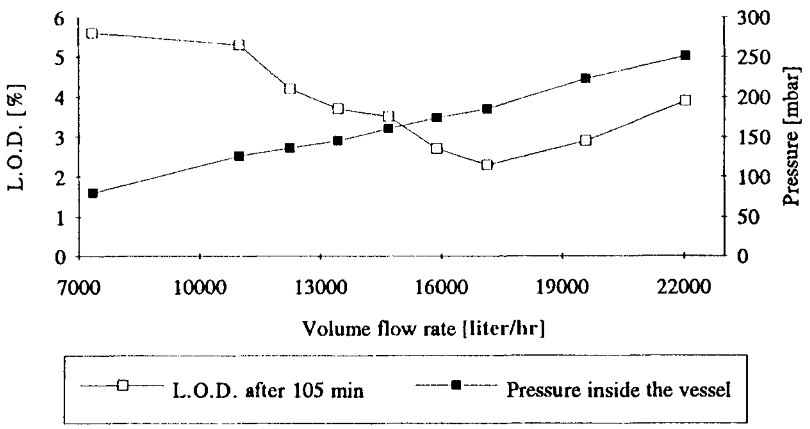

Figure 4. LOD and pressure level as functions of volume flow rate.

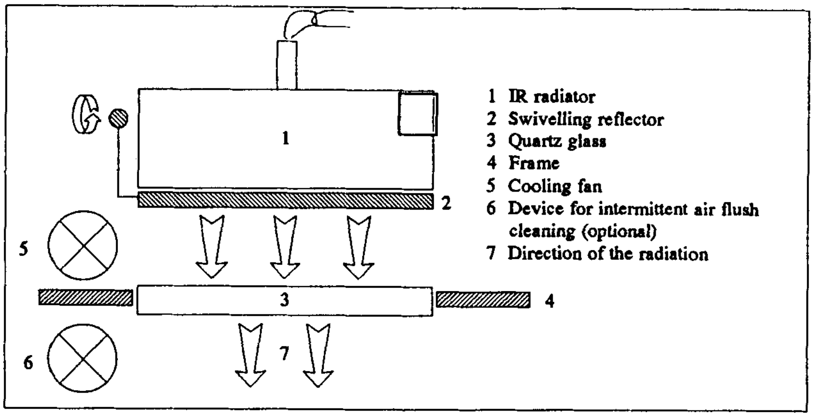

Figure 5. Installation of the IR equipment.

Using the Elstein ceramic radiator (1 in Fig. 5) it was necessary to apply a reflector (2, Fig. 5) between the quartz glass window and the radiator until the latter had reached its working temperature. Otherwise—since radiation is a function of temperature—the large part of its emitted energy would have been absorbed by the quartz glass. For the Heraeus glass tube radiator, a reflector was not required because its working temperature was reached within seconds. Cooling the quartz glass window properly avoided sticking of dust or granular particles to its inner surface when working with the tested material. Still, an additional device for intermittent air flush cleaning of the vacuum side surface of the quartz glass window would generally be desirable in assuring product and process safety.

Comparing the drying times of a trial without application of IR radiation (E 061121) to two other trials (E 121124, E 241122) with application of differently generated IR radiation while keeping all the other drying parameters constant, an evident difference could be observed (Fig. 6).

With both types of radiators a faster drying could be obtained than without additional IR radiation. From the beginning of the drying process this effect could be measured. Compared to the reference trial E 061121, drying time with the Heraeus system was about 30% shorter. The difference between the two radiator systems was mainly due to the available power of the individual radiator which could be emitted through the restricted area of the quartz glass window. While the power of the

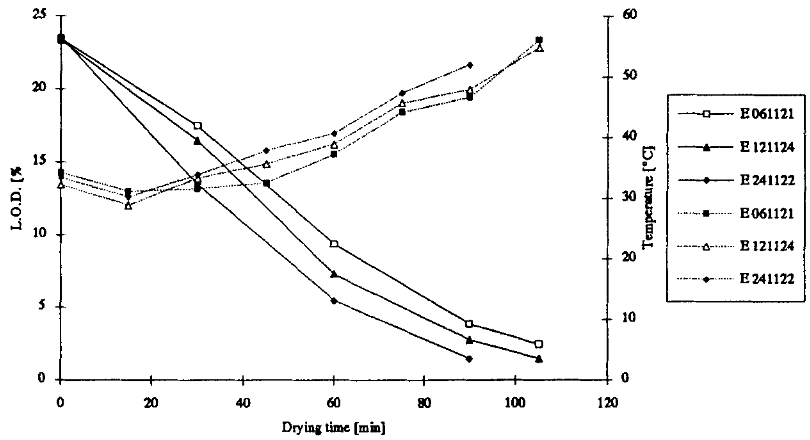

Figure 6. Drying curves and temperature of the granules with and without IR application [E 061121 (161 mbar, 60°C, GA.ST: 15,900 liter/hr, 230°C), E 121124 (160 mbar, 60°C, GA.ST: 15,900 liter/hr, 230°C, IR: Elstein), E 241122 (165 mbar, 60°C, GA.ST: 15900 liter/hr, 230°C, IR: Heraeus)].

Elstein radiator was 280 W the power of the Heraeus radiator was 920 W. In addition to the higher flux rate, the transmission behavior of the last-mentioned radiator is more favorable, because its emission spectrum fits better to the transmission characteristics of the quartz glass window (7).

While the IR runs showed an almost continuous increase of batch temperature after an initial phase, the reference trial had a plateau region between min 15 and 45, indicating a phase of constant drying rate (Fig. 6). Afterwards its temperature also increased continuously. Regarding thermal stress during drying, it seemed that at a corresponding LOD of 2.5% the stress exerted on the IR batches is significantly lower than in the reference trial. With this residual moisture content, the temperature of E 241121 was 47°C after 75 min drying and the temperature of E 121124 was 48°C after 90 min, whereas the temperature of E 061121 at the same moisture level was 56°C after 105 min. The explanation of this could be that together with the limited but sufficient penetration depth, radiation energy is absorbed by the moisture in the granules rather than by the solid phase.

Other observations supported this assumption. In a series of trials where only the Elstein radiator type was applied in comparison again with the reference trial E 061121, the temperature of the strip-gas was modulated while keeping the other drying parameters constant (Fig. 7). At the highest strip-gas temperature of 290°C (E 231121), only in the first 60 min of the drying process could a trend towards higher drying rates be registered. Compared to trial E 121124 with a strip-gas temperature of 230°C, no significant improvement could be obtained concerning drying times as the drying rates decreased at the end of the process. Just the opposite behavior could be seen in trial E 231124, where a strip-gas temperature of only 155°C was applied. During the first 60 min the drying rates were obviously worse than in the reference trial in spite of the use of IR radiation. After this time a surprising increase of drying rate could be observed which is supposed to be due to the penetrating effect of the IR radiation.

While the effect of the strip-gas flow seemed to be superior in the first part of the drying process because the surface of the granules was more or less saturated with moisture, this effect decreased in the following part of the drying process: There the use of IR radiation was more effective because of its ability to penetrate to the inner parts of the granules where the moisture was located, whereas the strip gas flow was only able to touch the already dry surface of the granules and had no direct contact with the wet zones. As in this phase, diffusion of the vapors and the moisture are the limiting factors concerning drying rate which cannot be influenced decisively by contact, convection, or vacuum

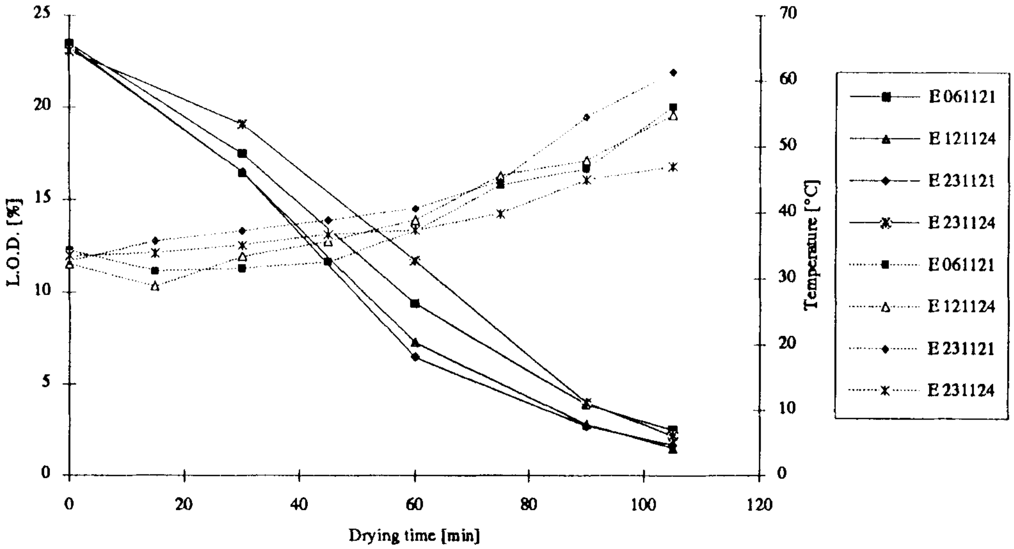

Figure 7. Drying curves and temperature of the granules with and without IR application [E 061121 (161 mbar, 60°C, GA.ST: 15900 liter/hr, 230°C), E 121124 (160 mbar, 60°C, GA.ST: 15900 liter/hr, 230°C, IR: Elstein), E 231121 (154 mbar, 60°C, GA.ST: 15900 liter/hr, 290°C, IR: Elstein), E 231124 (162 mbar, 60°C, GA.ST: 15900 liter/hr, 155°C, IR: Elstein)].

mechanisms any more (8), it is necessary to apply an additional drying principle which is able to interact directly with the moisture in the core of the granules and therefore must be able to penetrate the outer, already dried layers of the granular particles. Thus evaporation can take place and can be increased even in the very inner parts of the granules because the penetration depth of the applied IR radiation is greater than the average diameter of the particles (5).

Another benefit of granulation by the use of IR radiation was the reduced thermal stress (Fig. 7). Comparing trial E 231121 with 61°C to E 121124 with 55°C, after 105 min it was obvious that increasing strip-gas temperature over a critical limit did not increase the drying speed but only the temperature of the granules. On the other hand, the same drying result could be achieved with combined strip-gas and IR drying (E 231124), compared to strip-gas drying (E 061121), with less thermal stress (46°C vs. 56°C) due to reduced strip-gas temperature (155°C vs. 230°C).

Combined Vacuum, Contact, and Microwave Drying

In order to allow a direct comparison with a microwave-based drying process in the same scale, some experiments were carried out with a prototype Roto P 50 microwave processor. Concerning drying times, trials with a Collette Vactron 75 microwave processor (Machines Collette, B-2160 Wommelgem) had given satisfactory results before (2,4).

The parameters jacket temperature and magnetron power were varied. As could be expected, drying times were shortened by increasing the jacket temperature or the microwave power input. In trial E 250324 already after 15 min the temperature of the granules exceeded 40°C and increased up to 47°C at the end of the drying process (Fig. 8).

CONCLUSION

In order to develop an alternative drying process to microwave-based drying, some series of trials were carried out with a Zanchetta Roto P 50 pilot scale single-pot granulator.

1. Based on a standard granulation procedure, different combinations of drying mechanisms were evaluated. With the fundamental combination of vacuum and contact drying, only drying times of at least 3 hr could be realized (Fig. 8).

2. A significant reduction of drying times could be achieved by applying heated strip-gas. The positive effect of strip-gas application was due to the facts that the vapors were permanently removed by an unsaturated air stream (9), and that the particles were in direct contact with the strip-gas flow.

3. A further improvement of drying results was possible by application of IR radiation. Quartz tube radia-

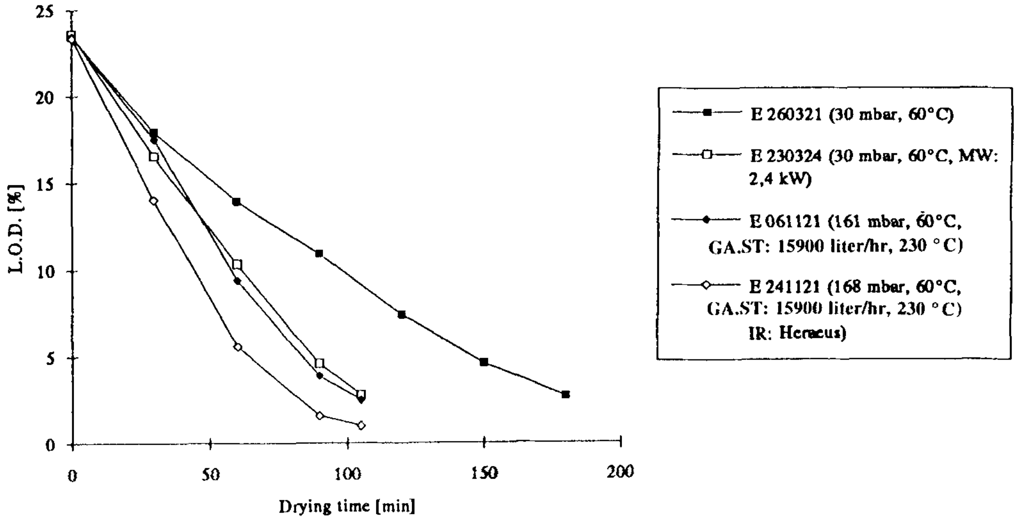

Figure 8. Synopsis of tested drying methods.

tors turned out to be superior to ceramic radiators as their higher energy flux and their more suitable emission spectrum gave a better energy transfer to the wet product. Especially in the end of the drying process, the small but sufficient penetration depth of IR radiation had a synergistic influence. The fact that its penetration depth is lower than that of microwaves is indeed an advantage if a permanent mixing of the directly irradiated surface of the batch is guaranteed. As the amount of absorption is inversely proportional to the wavelength, IR radiation is completely absorbed whereas the application of microwaves can lead to undesired effects such as local overheating or burning due to reflection and superposition of the microwaves, and maybe even breakdown of the electrical field (2,4,10). Compared to microwave technology, an IR-based drying process does not seem to involve such high risks for personnel and the product, and can be controlled easily.

Further investigation will be done in order to establish the new technology in larger production ranges.

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