

INVESTIGATION OF SPRAY DRYING PROCESSES FOR THERMOLABILE ENZYMES : A Systematic Approach

by

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CERTIFICATE

This is to certify that the thesis titled “**INVESTIGATION OF SPRAY DRYING PROCESSES FOR THERMOLABILE ENZYMES : A Systematic Approach**” submitted by **GYANA RANJAN SATPATHY** has been prepared under my supervision in conformity with rules and regulations of the “**INDIAN INSTITUTE OF TECHNOLOGY, DELHI**”. The research report and results presented in the thesis have not been submitted for any degree in any other university.

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Dedicated to

*Lord Shri Shri
Jagannātha, Baladeva
and Subhadrā Mahārānī*

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ABSTRACT

A comprehensive, state-of-the-art investigation of spray drying processes for thermolabile enzymes is carried out in this thesis. A rational and systematic approach to study spray drying processes is evolved, which helps considerably in design of experiments and leads to large savings in labor, time and cost. First, a sensitivity analysis of the spray drying operation on various operating parameters (e.g. inlet temperature (T_i), outlet temperature (T_o), liquid flow rate (\dot{m}_L) and air flow rate (\dot{m}_a)) is performed. The evaporative capacity of the spray dryer is determined under various operating conditions and safe regions of operation are recommended to ensure well-dried products. An inexpensive method is developed, tested and established to measure droplet sizes in a dense spray. In this innovative modification of the coated slide method, the actual droplets (and not their impressions) are visualized. Using this method, droplet sizes in the 3-200 micron range in a dense spray could be visualized reproducibly and with ease. The external-mixing two-fluid pneumatic atomizer is characterized and the droplet size distribution in the spray is experimentally measured as a function of the air and liquid flow rates and the physical properties (viscosity, surface tension, density) of the liquid. The Sauter mean diameter of these distributions is calculated by a computer program. A dimensional analysis is performed for two-fluid atomization and an empirical correlation is developed for prediction of Sauter mean diameter in terms of various dimensionless groups (Re , Oh , \dot{m}_G/\dot{m}_L). The empirical correlation is compared with correlations developed by other researchers. Next, spray-dried powder properties of various carrier and binder combinations are evaluated, first in the absence of enzyme (model system : alcohol dehydrogenase from baker's yeast) and later in its presence. More than 50 carrier-binder combinations are tried out and physicochemical properties of the spray-dried powder such as particle size and particle size distribution, angle of slide, angle of repose, angle of spatula, bulk density, compressibility, Kawakita constants, solubility, cohesion, moisture content and hygroscopicity are quantified.

The influence of inlet drying temperature and binder concentration on the powder properties is explored. The effect of powder particle size and particle size distribution on the powder properties is interpreted and discussed in detail.

Thermal inactivation of yeast alcohol dehydrogenase in the liquid state at various temperatures in the presence and absence of selected carriers and binders is studied. Nonlinear thermal inactivation behavior is observed and the kinetic parameters of the inactivation process are quantified using Nath's method. The thermostability of the enzyme is also evaluated using differential scanning calorimetry. The spray drying of alcohol dehydrogenase with selected carrier-binder combinations is investigated in detail. In addition to the indicators of powder quality listed earlier, retention of powder and retention of enzyme activity are also quantified. SDS-PAGE and total protein assays are carried out for validation. In the best cases, 100% enzyme activity retention and 190% powder retention is obtained. The powder morphology is evaluated by scanning electron microscopy. Economical, food-grade additives that yield spherical, non-hygroscopic spray-dried enzyme powder with outstanding flow properties, activity retention and shelf life are discovered. Spray drying of crude enzyme systems (cellulase, lipase) is also performed successfully. The shelf life of the spray-dried enzyme is studied at five different temperatures. The stability of spray-dried powder is found to be several hundred times greater than the stability in the liquid state.

Finally, a synthesis of all spray drying process stages is achieved. The *insitu dried* particle size is measured during the actual spray drying operation, With its help, the final agglomerated average dried particle size is predicted starting from the physicochemical properties of the liquid feed to the spray dryer. A mechanism of drying is proposed at the level of a single, representative droplet in the spray, Thus, a laborious, systematic and comprehensive experimental investigation of the spray drying process is successfully accomplished.

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