

Copyright is owned by the Author of the thesis. Permission is given for a copy to be downloaded by an individual for the purpose of research and private study only. The thesis may not be reproduced elsewhere without the permission of the Author.

## **Design and Development of a Modified**

# **Spouted Bed Coater for the**

# **Micro-encapsulation of Powders**

A thesis presented in partial fulfilment of the requirements for the degree of Master of Technology in Chemical Technology

Massey University

New Zealand

Mr Peter Andrew Bishop

2003

#### Abstract

A modified spouted bed coater was designed and constructed for the microencapsulation of solid particles. The coating of small particles with a polymer film can alter physical factors such as taste and release rate. These properties are particularly important in the field of pharmacology as the nature of the coating can be changed to prolong or target drug release based on physiological conditions such as pH and time.

The spouted bed coater was modified to induce gas and particle recirculation through a draft tube containing a venturi to increase droplet and particle mixing, while a high velocity gas jet and large diameter draft tube promotes the recirculation of gas and solid within the apparatus. The effectiveness of the design was tested in terms of gas and solid mass flows through the draft tube using a venturi within the draft tube and an induction detector to measure the mass flow.

To determine the effectiveness of the coater design in terms of coalescence and the influence of operational variables, a factorial experiment was conducted. The result of this experiment showed that the coalescence of particles was dominated by the relative humidity in the apparatus which was unable to be directly related to the operational variables.

The capacity to micro-encapsulate particles was demonstrated by coating fine table salt with an acrylic polymer Eudragit NE 40D in combination with bentonite clay as a free flow agent or glident. The results of this trial showed the distribution of polymer/clay and the reduction in dissolution rate as a function of particle size.

I

#### Acknowledgments

I would like to thank, my supervisor, Dr Jim Jones for the valuable guidance in the area of writing and for the opportunity to work on an exciting and challenging project. The technical staff in particulary, Russell Watson, Bruce Collins and John Edwards for there assistance in turning sketches into hardware. I would like to thank my wife, Janey and children, Claramae, Vanessa, Andrew and Caroline, for their support through the difficult times and the joy they bring to every day. My Thanks go also to my mother, Anne, and father, Derrick, for their encouragement over the years, with my struggle with dyslexia. The late Dr Jean Seabrook and SPELD for helping me find my potential.

## **Table of Contents**

1	Literatur	re Review
	1.1	Current Practices in Micro-encapsulation
		Liquid suspension micro-encapsulation
		Gas suspension methods
		Fluid bed processes
	1.2	Granulation and Encapsulation Mechanisms
		Viscous dissipation model
		Capillary suction models
		Conversion of Simons et al.[1994] model to give
		maximum collision velocity
		Comparison of viscous dissipation and capillary suction models 16
	1.3	Encapsulation Agents
		Latex coatings
	1.4	Prior Work
	1.5	Concluding Remarks
2	Design	
	2.1	Gas and Solids Entrainment
		Gas entrainment
		Entrained air
		Solids entrainment
	2.2	Atomizer Design
		Droplet diameter $D_{50}$ and $D_{90}$
	2.3	Drying and Dis-entrainment Chamber 44
		<i>Drying</i>
		Particle dis-entrainment
	2.4	Filter
	2.5	Instrumentation
		Sensors
		Pressure sensors

Temperature sensors
Humidity sensors
Gas flow sensors
Weight sensor
Induction sensor
Conditioning of sensor outputs 56
Analog to digital converter ADC 57
Computer system
Calibration
Pressure sensor calibration and conditioning
Calibration of critical flow nozzles
Conversion factors for voltage outputs to engineering units 61
Calculation of gas properties

3 Experimental	64
3.1 Mass Flow Rate of Solids	65
3.2 Gas Entrainment Rate	69
3.3 Particle Growth Rate Experiments	74
3.4 Micro-encapsulation	82
Measurement of NaCl extraction rate	84
4 Conclusions	87
References	90

## Nomenclature

а	particle radius, m
A	area of droplet, m <sup>2</sup>
$A_a$	area of atomizer air nozzle, m <sup>2</sup>
$A_{t}$	total area of atomizer air nozzle orifice, m <sup>2</sup>
С	rupture energy constant
$C_o$	capillary pressure of touching spheres, Pa
$C_D$	coefficient of drag
$d_p$	particle diameter, m
$d_{p(max)}$	maximum particle diameter for coalescence, m
$D_d$	diameter of droplet, m
$D_{dt}$	diameter of draft tube, m
$D_{j(0)}$	diameter of inlet gas jet at $z = 0$ , inlet gas nozzle diameter, m
$D_{50}$	50% of the mass of particles are less than this diameter, m
$D_{90}$	90% of the mass of particles are less than this diameter, m
$D_{max}$	maximum diameter of draft tube, m
dM/dt	rate of mass change, kg/s
е	coefficient of restitution
$F_{\gamma}$	surface tension force, N
$F_{sp}$	capillary suction force, N
$F_{\rm total}$	total liquid bridge force, $F_{\gamma}+F_{sp}$ , N
G	granule or particle growth rate , m/s
g	gravitational constant, m/s <sup>2</sup>
h	half the separation distance between particle surfaces, m
$h_c$	convection heat transfer coefficient, W m <sup>-2</sup> K <sup>-1</sup>
$h_0$	initial height of liquid layer on particle surface, m
$h_a$	height of surface asperity on surface of core particle, m
Н	height, m
$H_{min}$	minimum height of draft tube, m
$k_d$	thermal conductivity of droplet, W/mK
$K_f$	coefficient of resistance of fabric, kPa s/m
$K_p$	coefficient of resistance of powder, kPa m s/kg

$M_p$	individual particle mass, kg
$M_s$	total mass of solids in apparatus, kg
$M_{sf}$	mass of solids on filter fabric, kg
m	mass of particle, kg
Ν	number concentration, particles/m <sup>3</sup>
$N_{pixel}$	number of pixels
P	pressure, Pa
$Q_a$	mass of atomizer air flow, kg/s
$Q_l$	mass of atomizer liquid flow, kg/s
$Q_{g}$	mass flow of gas, kg/s
$Q_{g(Z)}$	mass flow of gas at point z on the z axis, kg/s
$Q_s$	mass flow of solids, kg/s
$Q_b$	mass flow of binder, kg/s
$r_{I}$	minimum radius of liquid bridge, m
$r_2$	radius of curvature of liquid bridge, m
t	time, s
t <sub>circ.</sub>	circulation time, s
$t_r$	gas retention time, s
u	velocity of particle, m/s
$u_0$	initial particle velocity, m/s
$u_{0(max)}$	initial maximum particle collision velocity for coalescence, m/s
U	velocity of gas, m/s
U <sub>t</sub>	settling velocity of particle, m/s
v <sub>r</sub>	radial particle velocity, m/s
$V_d$	volume of atomized droplet
$v_{rel}$	relative velocity, m/s
$\mathbf{V}_{\mathbf{p}}$	volume of particle, m <sup>3</sup>
$\mathbf{V}_{\mathbf{b}}$	volume of liquid bond, m <sup>3</sup>
v	velocity, m/s
W	work, J
x	particle separation distance, m
Z	distance from jet, m

### **Dimensionless Groups**

$b^*$	binder	to	solids	volume	ratio,	V <sub>b</sub> /V <sub>p</sub>	
-------	--------	----	--------	--------	--------	--------------------------------	--

- $b_{c}^{*}$  critical binder to solids ratio,  $(1+h_{a}/a)^{3}-1$
- $C_a$  Capillary number,  $\mu u/\gamma$
- *Nu* Nusselt number,  $h_d D_d / k_d$
- Re Reynolds number, U<sub>g</sub>D  $\rho_g/\mu_g$
- St<sub>v</sub> Stoke's number,  $2mu_o/3\pi\mu a^2$
- $V_b$  \* dimensionless bridge volume,  $V_b/a^3$
- $W^*$  dimensionless bond rupture energy, W/ya<sup>2</sup>
- ε dimensionless particle separation distance, 2h /a

#### Greek

α	contact angle, degrees			
β	half-filling angle, degrees			
$\Delta H_{vap}$	latent heat of vaporization, J/kg			
$\Delta T_{lm}$	log mean temperature difference, K			
μ	viscosity, kg/ms			
ρ	density, kg/m <sup>3</sup>			
$\rho_{s}$	density of solid, kg/m <sup>3</sup>			
γ	surface tension, N/m			
ε <sub>z</sub>	voidage of fluid bed at point z on the z axis			
θ	gas jet dispersion angle, degrees			
φ	the binder liquid contact angle			

 $\omega$  powder loading, kg/m<sup>2</sup>