

7844 A431

9361772

Particle Size Measurement

TERENCE ALLEN

Senior Consultant E.I. Dupont de Nemour and Company Wilmington, Delaware USA

FOURTH EDITION





E9361772



CHAPMAN AND HALL

LONDON ● NEW YORK ● TOKYO ● MELBOURNE ● MADRAS

UK Chapman and Hall, 11 New Fetter Lane, London EC4P 4EE

USA Chapman and Hall, 29 West 35th Street, New York NY10001

JAPAN Chapman and Hall Japan, Thomson Publishing Japan, Hirakawacho Nemoto

Building, 7F, 1-7-11 Hirakawa-cho, Chiyoda-ku, Tokyo 102

AUSTRALIA Chapman and Hall Australia, Thomas Nelson Australia, 480 La Trobe Street, PO Box 4725, Melbourne 3000

INDIA Chapman and Hall India, R. Sheshadri, 32 Second Main Road, CIT East, Madras 600 035

First edition 1968 Second edition 1975 Third edition 1981 Fourth edition 1990

© 1968, 1975, 1981, 1990 T. Allen

Typeset in 10/12pt Times by Best-set Typesetter Ltd, Hong Kong Printed in Great Britain by T.J. Press Ltd, Padstow, Cornwall

ISBN 0 412 35070 X

All rights reserved. No part of this publication may be reproduced or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording or otherwise, or stored in any retrieval system of any nature, without the written permission of the copyright holder and the publisher, application for which shall be made to the publisher.

British Library Cataloguing in Publication data

Allen, Terence

Particle size measurement. - 4th ed.

- 1. Particles. Size. Measurement
- I. Title II. Series

620.43

ISBN 0-412-35070-X

Library of Congress Cataloging-in-Publication Data Available

Particle Size Measurement

Powder Technology Series

EDITED BY BRIAN SCARLETT

Delft University of Technology The Netherlands

Many materials exist in the form of a disperse system, for example powders, pastes, slurries, emulsions and aerosols. The study of such systems necessarily arises in many technologies but may alternatively be regarded as a separate subject which is concerned with the manufacture, characterization and manipulation of such systems. Chapman and Hall were one of the first publishers to recognize the basic importance of the subject, going on to instigate this series of books. The series does not aspire to define and confine the subject without duplication, but rather to provide a good home for any book which has a contribution to make to the record of both the theory and the application of the subject. We hope that all engineers and scientists who concern themselves with disperse systems will use these books and that those who become expert will contribute further to the series.

Particle Size Measurement

T. Allen 4th edn, hardback (0 412 35070 X), 832 pages

Powder Porosity and Surface Area

S. Lowell and Joan E. Shields 2nd edn, hardback (0 412 25240 6), 248 pages

Pneumatic Conveying of Solids

R.D. Marcus, L.S. Leung, G.E. Klinzing and F. Rizk Hardback (0 412 21490 3), 592 pages

Particle Technology

Hans Rumpf Translated by F.A. Bull Hardback (0 412 35230 3), 216 pages

Acknowledgements

I would like to express my grateful thanks to Dr Brian H. Kaye for introducing me to the fascinating study of particle size analysis. My thanks are also due to numerous workers in this field for the helpful discussions we have had. Bradford University has provided me with a well-equipped laboratory in which, in teaching others, I have learnt some of the secrets of this science. One of my students was Mr T.S. Krishnamoorthy and the chapter on gas adsorption is taken from his M.Sc. thesis. At Bradford, Mr John C. Williams has always had the time to offer helpful advice and criticism. I make no apology for taking up so much of his time since his advice was invariably good and whatever virtue this book possesses is due, in part, to him.

My thanks are also due to holders of copyright for permission to publish and to many manufacturers who have given me full details of their products.

Finally, I would like to thank my wife for her forbearance while the writing of this book has been in progress.

Terence Allen

Preface to the fourth edition

Powder technology is a subject in its own right, and powder characterization is central to an understanding of this discipline.

In the eight years since the printing of the third edition of *Particle Size Measurement* there have been two big changes in my life. After thirty years of academia I have returned to industry, and after a lifetime in Great Britain I have emigrated to the United States.

In industry the initial demand is to relate powder properties to product performance and then to maintain powder consistency. This requires on-line or rapid off-line analysis which, in turn, has led to the demand for a whole range of new instruments whose primary function is process monitoring.

Historically, chemical engineering courses have concentrated on the behaviour of fluids, and engineers enter industry relatively unschooled in the subject of powder behaviour. Yet, when my colleagues Reg Davies and John Boughton surveyed three thousand Dupont products, they discovered that 80% involved powder at some stage of their manufacture. The results of this survey illustrate the need for more training in this key subject.

This edition reflects the changing image of powder characterization towards in-process size analysis. Hence the chapter covering on-line analysis has been largely re-written. Apart from this, I have expanded certain sections and describe the new instruments that have been introduced since the last edition.

The emphasis here is on commercial equipment and for an up-date on research and development in this area I recommend the reviews Particle Size Analysis by Barth, H.G. and Sun, S.T., *Anal. Chem.*, **57**, 151R, 1985 and 142R, 1987, and Critical Reviews in Analytical Chemistry by Miller, B.V. and Lines, R., **20**(2), 75–116, 1988.

Terence Allen

Senior Consultant E.I. Dupont de Nemours

Preface to the first edition

Although man's environment, from the interstellar dust to the earth beneath his feet, is composed to a large extent of finely divided material, his knowledge of the properties of such materials is surprisingly slight. For many years the scientist has accepted that matter may exist as solids, liquids or gases although the dividing line between the states may often be rather blurred; this classification has been upset by powders, which at rest are solids, when aerated may behave as liquids, and when suspended in gases take on some of the properties of gases.

It is now widely recognized that powder technology is a field of study in its own right. The industrial applications of this new science are far reaching. The size of fine particles affects the properties of a powder in many important ways. For example, it determines the setting time of cement, the hiding power of pigments and the activity of chemical catalysts; the taste of food, the potency of drugs and the sintering shrinkage of metallurgical powders are also strongly affected by the size of the particles of which the powder is made up. Particle size measurement is to powder technology as thermometry is to the study of heat and is in the same state of flux as thermometry was in its early days.

Only in the case of a sphere can the size of a particle be completely described by one number. Unfortunately, the particles that the analyst has to measure are rarely spherical and the size range of the particles in any one system may be too wide to be measured with any one measuring device. V.T. Morgan tells us of the Martians who have the task of determining the size of human abodes. Martian homes are spherical and so the Martian who landed in the Arctic had no difficulty in classifying the igloos as hemispherical with measurable diameters. The Martian who landed in North America classified the wigwams as conical with measurable heights and base diameters. The Martian who landed in New York classified the buildings as cuboid with three dimensions mutually perpendicular. The one who landed in London gazed about him despairingly before committing suicide. One of the purposes of this book is to reduce the possibility of further similar tragedies. The above story illustrates the problems involved in attempting to define the size of particles by one dimension. The only method of measuring more than one dimension is microscopy. However, the mean ratio of significant dimensions for a particulate system may be determined by using two methods of analysis and finding the ratio of the two mean sizes. The proliferation of measuring techniques is due to the wide range of sizes and size dependent properties that have to be measured; a twelve-inch ruler is not a satisfactory tool for measuring mileage or thousandths of an inch and is of limited use for measuring particle volume or surface area. In making a decision on which technique to use, the analyst must first consider the purpose of the analysis. What is generally required is not the size of the particles, but the value of some property of the particles that is size dependent. In such circumstances it is important whenever possible to measure the desired property, rather than to measure the 'size' by some other method and then deduce the required property. For example, in determining the 'size' of boiler ash with a view to predicting atmospheric pollution, the terminal velocity of the particle should be measured; in measuring the 'size' of catalyst particles, the surface area should be determined, since this is the property that determines its reactivity. The cost of the apparatus as well as the ease and the speed with which the analysis can be carried out have then to be considered. The final criteria are that the method shall measure the appropriate property of the particles, with an accuracy sufficient for the particular application at an acceptable cost, in a time that will allow the result to be used.

It is hoped that this book will help the reader to make the best choice of methods. The author aims to present an account of the present state of the methods of measuring particle size; it must be emphasized that there is a considerable amount of research and development in progress and the subject needs to be kept in constant review. The interest in this field in this country is evidenced by the growth of committees set up to examine particle size measurement techniques. The author is Chairman of the Particle Size Analysis Group of the Society for Analytical Chemistry. Other committees have been set up by The Pharmaceutical Society and by the British Standards Institution and particle size analysis is within the terms of reference of many other bodies. International Symposia were set up at London, Loughborough and Bradford Universities and it is with the last-named that the author is connected. The book grew from the need for a standard text-book for the Postgraduate School of Powder Technology and is published in the belief that it will be of interest to a far wider audience

Terence Allen

Postgraduate School of Powder Technology University of Bradford

Editor's foreword to the first edition

The study of the properties and behaviour of systems made up of particulate solids has in the past received much less attention than the study of fluids. It is, however, becoming increasingly necessary to understand industrial processes involving the production, handling and processing of solid particles, in order to increase the efficiency of such systems and to permit their control. During the past few years this has led to an increase in the amount of study and research into the properties of solid particle systems. The results of this effort are widely dispersed in the literature and at the moment much of the information is not available in a form in which it is likely to influence the education of students, particularly in chemical engineering, who may later be employed in industrial organizations where they will be faced with the prob, 'ms of solids' handling. It is also difficult for the engineer responsible for the design or selection of solids' handling equipment to make use of existing knowledge, with the result that industrial practice is not always the best that is achievable. It is hoped that the publication of a series of monographs on Powder Technology, of which this is the first, will help by providing accounts of existing knowledge of various aspects of the subject in a readily available form.

It is appropriate that the first monograph in this series should deal with the measurement of the size of small particles since this is the basic technique underlying all other work in powder technology. The reliability of research results, for example, on the size reduction of solid particles, cannot be betted than the reliability of the particle size measurement techniques employed. Too often the difficulties and limitations of size measurement are ignored in such work, so that any conclusions become suspect. The importance of a thorough understanding of the problems involved in measuring the size of small particles for anyone working in any aspect of powder technology is therefore difficult to overestimate. It is hoped that this monograph, written by an experienced size analyst who has studied critically most of the methods described, will be of value in encouraging an informed and critical approach to the subject and that it will help in the selection of equipment and in realistic assessment of the value of particle size measurements.

Editor's foreword to the fourth edition

This book has now reached its fourth edition and is undoubtedly the standard reference book on particle size measuring techniques. The book started life as a short course text and, in its successive editions, it has been polished and extended to become a balanced and comprehensive text, full of information and reflecting great experience. This book is the flag hip of the series and I hope that many other books will follow it to the same level of maturity.

Brian Scarlett

January 1990

9361772

Contents



Ackn	owledgements	xviii	
Prefa	ce to the fourth edition	xix	
Prefa	ce to the first edition	XX	
Edito	or's foreword to the first edition	xxii	
Edito	or's foreword to the fourth edition	xxiii	
1	Sampling of powders	1	
1.1	Introduction	1	
1.2	Theory	2	
1.3	Weight of sample required	2 5	
1.4	Statistical considerations	7	
1.5	Golden rules of sampling	8	
1.6	Bulk sampling	9	
	1.6.1 Stored non-flowing material	9	
	1.6.2 Stored free-flowing material	9	
	1.6.3 Moving powders	10	
	1.6.4 Sampling from a moving stream of powder	10	
	1.6.5 Sampling from a conveyor belt or chute	13	
	1.6.6 Sampling from a bucket conveyor	16	
	1.6.7 Bag sampling	16	
	1.6.8 Sampling spears	16	
	1.6.9 Sampling from wagons and containers	19	
	1.6.10 Sampling from heaps	21	
1.7	Slurry sampling	23	
1.8	Sample dividing	23	
	1.8.1 Scoop sampling	25	
	1.8.2 Coning and quartering	29	
	1.8.3 Table sampling	29	
	1.8.4 Chute splitting	29	
	1.8.5 The spinning riffler	31	
1.9	Miscellaneous devices	33	
1.10	Reduction from laboratory sample to analysis sample	34	
1.11			
1.12	Experimental tests of sample-splitting techniques	37 38	

vi Contents

2	Sampling of dusty gases in gas streams	41
2.1	Introduction	41
2.2	Basic procedures	44
	2.2.1 Sampling positions	44
	2.2.2 Temperature and velocity surveys	45
	2.2.3 Sampling points	46
2.3	Sampling equipment	47
	2.3.1 Nozzles	47
	2.3.2 Dust-sampling collector	50
	2.3.3 Ancillary apparatus	57
	2.3.4 On-line dust extraction	57
	2.3.5 The Andersen stack sampler	58
2.4	Corrections for anisokinetic sampling	60
2.5	Probe orientation	66
2.6	Radiation methods	67
3	Sampling and sizing from the atmosphere	72
3.1	Introduction	72
3.2	Inertial techniques	76
3.3	Filtration	87
3.4	Electrostatic precipitation	88
3.5	Electrostatic charging and mobility	90
3.6	Thermal precipitation	92
3.7	The quartz microbalance	95
3.8	Optical sensing zone methods	97
	3.8.1 Air Technology	102
	3.8.2 Atcor Net 2000 3.8.3 Bausch and Lomb	102
		102
	3.8.4 Beckman	103
	3.8.5 Centre for Air Environmental Studies	103
	3.8.6 Climet Series 7000	103
	3.8.7 Coulter Model 550 contamination monitor	103
	3.8.8 Dynac	104
	3.8.9 Gardner	104
	3.8.10 G.C.A. Miniram	104
	3.8.11 Insitec PCSV-P	104
	3.8.12 Kratel Partoscope	105
	3.8.13 Leitz Tyndalloscope	105
	3.8.14 Met One particle counters	105
	3.8.15 Pacific Scientific Hiac/Royco particle counting systems	106
	3.8.16 Particle Measuring Systems	106
	3.8.17 RAC particle monitors	107
	3.8.18 Rotheroe and Mitchell digital dust indicator	108
	3.8.19 Saab photometer	108

) Sartorius	108
		Sinclair	108
		2 Techecology	109
		3 TSI particle counters	109
		The particulate volume monitor	109
3.9	Cond	ensation nucleus counters	110
3.10		sion battery	110
3.11	The a	erodynamic particle size analyser	112
3.12	Misce	llaneous techniques	114
4		ele size, shape and distribution	124
4.1	Partic	le size	124
4.2	Partic	le shape	128
		Shape coefficients	129
		Shape factors	132
	4.2.3	Applications of shape factors and shape coefficients	135
	4.2.4	Shape indices	140
	4.2.5	Shape regeneration by Fourier analysis	141
	4.2.6	Fractal dimension characterization of textured surfaces	142
4.3	Deter	mination of specific surface from size distribution data	144
		Number distribution	144
	4.3.2	Surface distribution	144
	4.3.3	Volume distribution	145
4.4	Partic	le size distribution transformation between number,	
	surfac	e and mass	145
4.5		nge diameters	147
4.6		le dispersion	153
4.7	Metho	ods of presenting size analysis data	153
4.8	Devic	es for representing the cumulative distribution curve as a	
	straigl	ht line	156
		Arithmetic normal distributions	156
	4.8.2	The log-normal distribution	159
	4.8.3	The Rosin-Rammler distribution	163
	4.8.4	Mean particle sizes and specific surface evaluation for	
		Rosin-Rammler distributions	164
	4.8.5	Other particle size distribution equations	164
	4.8.6	Simplification of two-parameter equations	165
	4.8.7	Evaluation of non-linear distributions on	
		log-normal paper	166
	4.8.8	Derivation of shape factors from parallel	
		log-normal curves	169
4.9	The la	w of compensating errors	170
4.10		native notation for frequency distribution	173
	4.10.1	Notation	174

viii Contents

	4.10.2 Moment of a distribution	174
	4.10.3 Transformation from $q_t(x)$ to $q_r(x)$	174
	4.10.4 Relation between moments	175
	4.10.5 Means of distributions	176
	4.10.6 Standard deviations	177
	4.10.7 Coefficient of variation	177
	4.10.8 Applications	177
	4.10.9 Transformation of abscissa	179
4.11	Phi-notation Phi-notation	181
4.12	Manipulation of the log-probability equation	182
	4.12.1 Average sizes	183
	4.12.2 Derived average sizes	185
	4.12.3 Transformation of the log-normal distribution by	
	count into one by weight	186
4.13	Relationship between median and mode of a log-normal	
	distribution	187
4.14	An improved equation and graph paper for log-normal	
	evaluations	187
	4.14.1 Applications	189
5	Sieving	192
5.1	Introduction	192
5.2	Woven-wire and punched plate sieves	193
5.3	Electroformed micromesh sieves	194
5.4	British Standard specification sieves	197
5.5	Methods for the use of fine sieves	198
	5.5.1 Machine sieving	199
	5.5.2 Wet sieving	200
	5.5.3 Hand sieving	202
	5.5.4 Air-jet sieving	203
	5.5.5 The sonic sifter	204
	5.5.6 Felvation	200
	5.5.7 Self-organized sieve (SORSI)	201
5.6	Sieving errors	208
5.7	Mathematical analysis of the sieving process	210
5.8	Calibration of sieves	213
6	Microscopy	217
6.1	Introduction	217
6.2	Optical microscopy	21
	6.2.1 Sample preparation	219
	6.2.2 Particle size distributions from measurements on	
	plane sections through packed beds	22
63	Particle size	22

6.4	Transmission electron microscopy (TEM)	223
	6.4.1 Specimen preparation	224
	6.4.2 Replica and shadowing techniques	226
	6.4.3 Chemical analysis	227
6.5	Scanning electron microscopy (SEM)	227
6.6	Manual methods of sizing particles	228
	6.6.1 Graticules	229
	6.6.2 Training of operators	232
6.7	Semi-automatic aids to microscopy	233
6.8	Automatic counting and sizing	240
6.9	Automatic image analysis	241
6.10	Specimen improvement techniques	243
6.11	Statistical considerations governing the determination of size	
	distributions by microscope count	243
	6.11.1 Frequency distribution determination	243
	6.11.2 Weight distribution determination	244
6.12	Conclusion	245
7	Interaction between particles and fluids in a gravitational field	249
7.1	Introduction	249
7.2	Relationship between drag coefficient and Reynolds number	
	for a sphere settling in a liquid	251
7.3	The laminar flow region	252
7.4	Critical diameter for laminar flow settling	253
7.5	Particle acceleration	254
7.6	Errors due to the finite extent of the fluid	255
7.7	Errors due to discontinuity of the fluid	257
7.8	Brownian motion	258
7.9	Viscosity of a suspension	261
7.10	Calculation of terminal velocities in the transition region	261
7.11	The turbulent flow region	266
7.12	Non-rigid spheres	266
7.13	Non-spherical particles	267
	7.13.1 Stokes' region	267
	7.13.2 The transition region	270
7.14	Concentration effects	272
7.15	Hindered settling	278
	7.15.1 Low-concentration effects	278
	7.15.2 High-concentration effects	279
7.16	Electro-viscosity	280
8	Dispersion of powders	285
8.1	Discussion	285
8.2	Theory of wetting	286

x Contents

8.3 8.4	The use of glidants to improve flowability of dry powders	293
	Density determination	293
8.5	Viscosity	297
8.6	Sedimentation systems	298
8.7	Densities and viscosities of some aqueous solutions	303
8.8	Standard powders	304
9	Incremental methods of particle size determination	310
9.1	Basic theory	310
	9.1.1 Variation in concentration within a settling suspension	210
	9.1.2 Relationship between density gradient and	310
	concentration	311
9.2	Resolution for incremental methods	312
9.3	The pipette method	313
	9.3.1 Experimental errors	317
9.4	The photosedimentation technique	320
	9.4.1 Introduction	320
	9.4.2 Theory	321
	9.4.3 The extinction coefficient	323
	9.4.4 Photosedimentometers	325
	9.4.5 Discussion	327
9.5	X-ray sedimentation	328
9.6	Hydrometers	332
9.7	Divers	335
9.8	The specific gravity balance	336
9.9	Appendix: Worked examples	337
	9.9.1 Wide-angle scanning photosedimentometer: analysis	
	of silica	337
	9.9.2 Conversion from surface distribution to weight	
	distribution	338
	9.9.3 The LADAL X-ray sedimentometer: analysis of	
	tungstic oxide	339
10	Cumulative methods of sedimentation size analysis	344
10.1	Introduction	344
10.2	Line-start methods	344
10.3	Homogeneous suspensions	345
10.4	Sedimentation balances	346
	10.4.1 The Gallenkamp balance	349
	10.4.2 The Sartorius balance	351
	10.4.3 The Shimadzu balance	353
	10.4.4 Other balances	353
10.5	The granumeter	355