

# THE TECHNOLOGY OF POWDER COATINGS

by

S. T. HARRIS B.Sc.

Edited by

E. C. ROBERSON Ph.D., B.Sc., A.R.I.C.

PORTCULLIS PRESS LONDON

First published in 1976 by Portcullis Press Ltd.

Queensway House, 2 Queensway, Redhill, Surrey, RH1 1QS

© Portcullis Press Limited

ISBN 90199493 6

All rights reserved. No part of the Publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording or otherwise without the prior permission of Portcullis Press Limited

A Portcullis Press Publication

# THE TECHNOLOGY OF POWDER COATINGS

#### **Preface**

At their present stage of development, powder coatings must seem to the newcomer to be a confusion of plastics and paint technologies. I have, therefore, endeavoured to present a factual account of the techniques currently used in the industry and, where possible, to highlight the aspects which need to be developed.

The technology of powder coatings will alter considerably during the next decade but it will surely become established as a technology in its own right. In compiling this account I am deeply indebted to the suppliers of raw materials, manufacturing machinery and application and testing equipment who have assisted me with descriptions of their products and photographs to illustrate the text.

A list of these suppliers is given in the final section of the book to enable the reader to obtain up-to-date information on the products described in the text.

The manner in which this book is presented owes much to the careful editing of Dr E. C. Roberson and I am extremely grateful to him for easing the burden on a first time author.

The Old Rectory Nether Whitacre Near Coleshill West Midlands S. T. Harris August 1976

#### **Foreword**

Powder coatings designed for use in industry, have now reached an acceptance level at which they can be regarded as a viable form of industrial finishing.

There comes a time in any new technology such as this when a considerable amount of information is available, acquired both by ad hoc methods and by organised research, but when no one has either enough knowledge or the dedication to put this information into a collected form and so allow both the practitioner and the newcomer to the industry to understand the status which has been reached and the possibilities for the future.

The powder coating industry in its present form has grown in a remarkable way in the last ten years. Appreciable quantities of thermoplasts were used even ten years ago, when the thinner self-adherent coatings, offered by the thermoset powders, were just coming into use. In those early days they were used mainly to give special protective properties, improved resistance to wear and damage or because of their good electrical properties, but their appearance in general was rather poor. Tremendous efforts by resin manufacturers, powder formulators and manufacturers together with enormous improvements in electrostatic spraying equipment, have now made these thermoset powder-coatings comparable in appearance with the best of the liquid coatings while still offering considerable advantages in performance.

Other factors, such as the enormous increase in recent years in the cost of hydrocarbon and organic solvents generally, have made powder coatings compare favourably in cost with liquid coatings. Environmental, safety and health considerations also make them increasingly attractive to manufacturers who need to apply coatings for protective and aesthetic reasons.

The author, whom I have known personally for many years, has a life long experience in the surface coatings field. This has included in recent years a period of dedication to powder coatings. Not only a scientist and a formulator of powder coatings, he has also had a wealth of experience of manufacture, setting up two plants for this purpose, the second being his own company.

It is therefore with considerable pleasure that I commend this book to whoever reads it. It is a fascinating study of the development of a new technology, done with considerable care and devotion and presented in a

clear and organised manner. The fact that powder technology has still a long way to travel in no way detracts from this book, which should remain a standard book of reference for years to come.

**Drynamels Limited** 

F. C. J. Ruzicka
Ph.D., D.I.C., A.R.C.S., F.R.I.C.
Managing Director

### **Contents**

PREFACE	v
FOREWORD	vii
INTRODUCTION	1
THE CHEMISTRY AND MANUFACTURE OF POWDER	
COATING RESINS	12
THE CROSS-LINKING REACTIONS OF THERMOSET POW-	
DER-COATING RESINS	28
THE FORMULATION OF POWDER COATINGS	43
MANUFACTURING PROCESSES — SUMMARY OF CHAP-	
TERS 5, 6, 7 and 8	59
PREMIXING	60
HOT MELT COMPOUNDING OF POWDER COATINGS	77
GRANULATION AND FINE GRINDING PROCESSES	103
THE POWDER PARTICLE	122
POWDER COATINGS APPLIED BY FLUIDIZED BED	154
POWDER COATINGS APPLIED BY ELECTROSTATIC SPRAY	170
SAFETY PROCEDURES	207
TESTING PROCEDURES	231
DESIGN AND LAYOUT OF POWDER MANUFACTURING	
PLANT	251
DESIGN AND LAYOUT OF POWDER COATING INSTAL-	
LATIONS	268
FUTURE DEVELOPMENTS	286
APPENDIX I List of manufacturers	293
APPENDIX II Technical Data	298
INDEX	208

## List of Tables

Table 1	C	_
Table 1.	Consumptions of ingredients	7
Table 2.	Acrylic functional groups	14
Table 3.	The effect of varying epoxy resin components	16
Table 4.	Commercially available epoxy resins	17
Table 5.	Some commercially available curing agents for epoxy	
•	powder coatings	36–37
Table 6.	Schematic illustration of powder coating production	n
	procedures	59
Table 7.	Range of drum hoop mixers	63
Table 8.	Range of drum tumbling machines	63
Table 9.	Range of oblique cone blenders	65
Table 10.	Range of double Z-blade mixers	79
Table 11.	Range of Buss Ko-Kneaders	85
Table 12.	Dimensions of planetary extruders in mm	88
Table 13.	Technical data of planetary extruders	89
Table 14.	Drawing of standard cooler with technical data and	i
	••	100-101
Table 15.	Specification of the Micron separator N type	137
Table 16.	Range of Micron Separators	139
Table 17.	The shape factor	158
Table 18.	The metal factor	160
Table 19.	Relative performances of commercially available thermo	
	setting powder coatings	248_9

#### 1

#### Introduction

#### SUMMARY

In these days of escalating raw material prices, and a worldwide shortage of the essential components required by the Paint Industry, it is not surprising that the new techniques, which improve the overall efficiency of painting methods, should now be considered and introduced as replacements for many conventional industrial finishing processes.

Already the new techniques that have been introduced on an industrial scale include electrophoretic painting, the use of water borne primers and top coats, and non-aqueous dispersions (NAD). These methods have one common purpose, namely, to reduce the dependence of organic coatings upon high proportions of costly organic diluents and solvents which have to be wasted in the drying process. The need to improve coating efficiency and to reduce the volatile content of paints has also been prompted by the desire to reduce environmental pollution caused by the disposal of these organic wastes.

It is little more than ten years since spray applied powder coatings were introduced, and it is this technical development which is fully described in the following pages although reference will be made, from time to time, to comparative assessments of powder coatings and conventional paints.

#### HISTORICAL BACKGROUND

Thermosetting powder coatings were introduced in the United States during the late 1950's. They were relatively simple products, dry blends of epoxy resin, pigments and hardener, dispersed by ball milling techniques. The fluidized bed method for applying thermoplastic powders was already well established at the time and the new thermosetting products were initially applied in the same manner. But due to the heterogeneous nature of the product, the individual components were easily classified on a size and weight basis in the fluidized bed and the results were inconsistent. At this stage of development thermoset powders were not considered as potential decorative coatings, but rather as chemically resistant or electrically insulating coatings with film thicknesses, in excess of two hundred microns.

In the early 1960's the Shell Research laboratories at Egham began the first serious investigation into better methods of compounding these products, and they described two methods for formulating thermosetting epoxy based powder coatings. In the first method, liquid epoxy resins and liquid hardeners were reacted to produce a partially cured B stage in which the liquid components were polymerised to a low melting point solid which could then be comminuted to a fine powder. The fully cured C stage was achieved by stoving the B stage powders at high temperature. It was, however, extremely difficult to ensure good batch to batch reproducibility, for the properties of the B stage were sensitive to small temperature variations and at low temperatures the reaction would take an excessively long time to proceed to the B stage. The most serious drawback however was the exotherm which could occur when the compounded materials were cast in too thick a laver, resulting in rapid cross linking to form the C stage. These products were also characterized by short shelf life. While B stage powders are no longer produced commercially for the reasons stated, it must be said that thickness for thickness they gave cured films of exceptional chemical resistance which cannot be matched by current powder coating systems.

The second method investigated by the Shell laboratories was termed the fusion process where solid epoxy resins, pigments and curing agent (dicyandiamide) were compounded in a heated Z-blade mixer. This compounding process was an immense step forward in the development of thermosetting powder coatings but it still had certain major faults. It was essentially a batch production process requiring excessive cleaning operations between batches, and the performance of the finished product was limited by the low reactivity of the systems manufactured by Z-blading. The use of heat activated curing agents, such as boron trifluoride complexes was partly successful in producing systems curing at lower temperatures but the problems of cleaning between batches is still a major restriction to the use of the Z-blade mixer.

Continuing research at Shell resulted in 1964 in the introduction of extrusion methods and it is this process which is currently favoured for the large scale continuous compounding of powder coatings. The high cost of the equipment deterred many powder manufacturers and the small paint producer, contemplating his own powder production facility, often proceeded no further than obtaining the quotation for the extruder.

The application of powder coatings by spray methods to non-preheated articles became a reality in 1962 with the introduction by S.A.M.E.S. of electrostatic powder spray guns. Early and cumbersome hand operated equipment was progressively improved to give light-weight guns which could be easily automated, and many novel methods for the electrostatic application of powder coatings are to be found in recent patent literature. Rapid development in application equipment over the past five years, and the gradual acceptance by industry that powder coating must be considered as a separate process from conventional painting methods, have been the main factors in the rapid growth in powder production over this period.

#### TYPES OF POWDER COATING

There are two main classes of powder coating; the thermoplastic and the thermosetting types. The resins used in both types have been employed in solution form for many years by the Paint Industry, and a number of the more successful coatings have resulted from the combination of compatible resins of both series.

Thermoplastic resins are generally characterized as materials of comparatively high molecular weight which exhibit excellent chemical resistance qualities, toughness and flexibility. Thermosetting resins are initially of lower molecular weight and a high molecular weight complex structure is built up during the curing cycle. As low molecular weight materials they show better flow properties, good surface wetting characteristics and hence, excellent adhesion to the substratum.

Traditionally, thermoplastic resins were well established in the field of powder coatings before thermoset products were developed. In the fluidized bed method of application, thermoplastic powders would melt in contact with the heated article to be coated, and flow out to give a continuous film upon cooling. Spoiled articles could be repaired by reheating and reflowing the film. However, application by this technique resulted in film thicknesses greater than 250 microns and the high raw material costs of some resins made this process of interest only for specialized applications. In the electrical industry a considerable tonnage of thermoplastic powder coating is used for electrical insulation purposes, a field which continues to grow at a healthy rate.

High molecular weight resins exhibiting toughness and flexibility are difficult to reduce in particle size without resorting to the costly techniques of cryogenic fine grinding. It is this factor which makes it difficult to process thermoplastic resins, such as nylon, to the particle size suitable for thin film electrostatic spray application.

Thermoplastic resins suitable for use in powder coatings include polyvin'yl chloride, polyvinyl butyral, nylons, polyesters, acrylics and certain high molecular weight epoxies.

The list of thermosetting resins suitable for use in powder coatings includes heat convertible forms of many in the thermoplastic class but one resin alone is predominant, namely the epoxy resin. More than 90% of all thermosetting powder coatings are based upon the epoxy resin, and it is predicted that this domination will continue for several years.

In the period of development since 1970, polyester resins have been introduced as alternatives to epoxy resins, and acrylic resins are also available.

There are several reasons why epoxy resins are widely used in powder coatings. In the development era they already existed as coating resins possessing relatively low melting points with glass transition temperatures high enough to prevent the fine particles agglomerating at room temperatures. Their most important attributes are excellent adhesion and very good

chemical resistance. Epoxy resins are capable of reacting with a wide range of organic compounds so that particular film properties may be selected by the correct choice of curing agent.

Epoxy resins are open to criticism with respect to heat and light stability and they also exhibit a tendency to chalking on exposure outdoors. Polyester resins and acrylics show distinct advantages over the epoxies in their improved heat and light resistance qualities and some of these resins are markedly better in respect of exterior weathering properties.

It is possible to cross-link hydroxyl-containing resins with an isocyanate to produce a polyurethane coating. This method, however involves the use of a blocked isocyanate in which the volatile component is driven off during the stoving operation. This volatile content defeats the main objective of powder coatings which should be the attainment of a 100% non-volatile system.

Thermosetting vinyl systems have also been developed and phenolic resins may be used in conjunction with epoxy resins. Amino resins may be included as co-reactants with polyesters and acrylics.

Almost daily, new resins are developed for use in powder coatings and the formulating techniques of the paint technologist will, no doubt, be readily applied to powder coatings.

#### MARKET FOR POWDER COATINGS

During the period 1969–1976 there have been many conferences dealing with powder coatings and inumerable statistics have been presented to illustrate the growth rate in this new market. While these figures have at times appeared to be contradictory there is no doubt that considerable growth has occurred during this period. In the early 1970's an annual growth rate of 25–30% was reported in many countries. This rate has now increased to between 50 and 100% and it is expected to maintain this for the next 5–6 years.

The assessment of the market potential must begin with an appreciation of the factors which are responsible for this high growth rate:

- (a) In the United States and Japan, industry must comply with the terms of stringent anti-pollution legislation, while in all countries there is a growing awareness of the need to protect the environment. Powder coatings cause virtually no atmospheric pollution, nor do they produce effluent. In fact they are generally free from any toxic hazard.
- (b) The current shortages of all chemicals derived from petroleum, and the escalating price of paint solvents, make it all the more desirable to consider formulating paints which do not require solvents either during manufacture or application.
- (c) There are distinct advantages to be gained, both financially and on the production line by the use of a one coat dry coating to replace a two coat wet process.

- (d) The fluidized bed is a simple process to operate, and the ease of application of powder coatings by electrostatic spray to complex articles removes the need to employ highly skilled applicators.
- (e) Application processes for powder coatings are easy to automate and the costs of equipment are reasonable.
- (f) New developments with polyester, polyurethane and acrylic resins are increasing the scope of powder coatings.
- (g) The fire hazard associated with wet paint methods is considerably reduced.
- (h) In practice, the efficiency of usage of powder coatings in automated plants is 95-98%.

Manufacturers who have introduced powder coatings have invariably recovered the capital cost of the plant within three years by the considerable savings effected in the following areas:

- 1. Primer coats are eliminated in most cases.
- 2. Overspray losses are greatly reduced.
- 3. Labour costs may be cut by as much as 50%.
- 4. Cleaning of the wet paint booth is eliminated.
- 5. Powder coatings are supplied ready for use and do not require thinning or mixing before application.
- 6. Factory or paint shop heating costs are reduced for it is not necessary to exhaust large quantities of warm air from the plant.
- 7. Reject rates are low.
- 8. The plant occupies less space since there is no flash-off zone and the oven may be reduced in size.
- 9. Costs of paint storage may be reduced.
- 10. Dust proof rather than flame proof equipment is specified.
- 11. Insurance rates should be lower, although this does not always apply when a new process is assessed by insurers.

#### The Size of the Market

A world wide consumption of powder coatings of all types has been estimated by De Bell and Richardson. (1)

1971 1976 1980 Total usage (metric tonnes) 44,500 105,000 208,000

An assessment of the position in Japan was given by Hikasa. (2)

 1971
 1972
 1973
 1974
 1975

 Usage (metric tonnes)
 1,200
 2,400
 3,700
 6,100
 9,000

Even this significant growth will only account for 0.6% of the total Japanese paint sales by 1975.

It has been predicted by Kut<sup>(3)</sup> that all types of powder coatings applied by electrostatic spray will show the following growth based on the 1970 figures for Europe.

	1970	1975	(Metric tonnes)
West Germany	1,300	5,000	
France	800	4,000	
Italy	500	3,500	
Benelux	400	1,500	
Others	400	3,000	
U.K.	200	3,000	

Bruniere<sup>(4)</sup> has published comparative sales of thermoplastic powders compared with thermosetting powder. In 1972 there were 43,000 tonnes of powder coatings sold through the world and of this figure 33,000 tonnes were thermoplastic while 10,000 tonnes were thermoset. The breakdown of thermoplastic sales is reported as:

Polyethylene	21,000 tonnes
PVC	7,500 tonnes
Polyamides	2,000 tonnes
Fluorocarbons	1,800 tonnes
Others	1,600 tonnes

#### Market Estimates and Predictions for the U.K.

These figures relate to resin and finished product sales and the predictions are entirely dependent upon availability of the resins. They do not include the considerable tonnage of thermoplastic powder applied by fluidized bed techniques.

The eventual size of the 1978 market will be dictated by the acceptance of powder coatings into the automotive and domestic appliance finishing fields.

Epoxy resins will continue to dominate the market up to 1976 when their sales are expected to level out at approximately 2,000 tonnes per annum.

It is difficult to separate the potential markets for polyester and acrylic powders for they are, to some extent, interchangeable, and resin costs may dictate the eventual choice between these two.

The sale of powder coatings in 1973 was divided among the following industries:

Metal furniture	140 tonnes
Domestic appliance	114 tonnes
Automotive	76 tonnes
Exterior quality	42 tonnes
Electrical	147 tonnes
General engineering	372 tonnes
Trade coaters	112 tonnes

The technical requirements of each industry were:

#### INTRODUCTION

Table 1. Consumptions of Ingredients

	Epoxies	Polyesters	Acrylics
	Resi	ins (tonnes)	
1968	30		
1969	50	_	_
1970	70	_	_
1971	140		
1972	250	2	2
1973	550	50	3
1974	900	120	10
1975	1,400	260	100
1976	1,800	700	700
1977	2,000	1,000	1,000
1978	2,200	1,800 +	1,800 +
	Powder o	coatings (tonnes	)
1968	50		_
1969	90		_
1970	120		-
1971	250	_	
1972	450	3	2
1973	940	60	3
1974	1,500	150	10
1975	2,500	350	170
1976	3,100	1,000	1,000
		4 400	1 400
1977	3,500	1,400	1,400

#### METAL FURNITURE

Major outlets are: schools, offices and factory canteens.

#### Properties

Good adhesion to phosphated mild steel with scratch resistance in excess of 4,000 gm.

Flexibility and adhesion characteristics—to withstand both direct and reverse impact.

Mainly semi-gloss finish.

Film thickness, in one coat, of 63 to 76 microns ( $2\frac{1}{2}$  to 3 thousandths of an inch). Corrosion resistance requirements are restricted to garden furniture applications.

#### DOMESTIC APPLIANCES

Major outlets are: deep-freeze units, refrigerators, water heaters, spin drier cabinets, plastic iron handles, radiators.

#### **Properties**

Good adhesion, scratch resistance in excess of 4,000 gm.

High gloss and good flow.

Mainly white or pastel shades.

Light and heat stability properties may only be provided by acrylic or polyester powders.

Stain resistance qualities are met by epoxy powders.

Good alkali and humidity resistance in excess of 2000 hrs. Epoxy powders are most suitable.

Substratum may be mild steel, zinc coated steel or phenolic plastic.

Outlets in the food industry may require the coatings to have F.D.A. status.

#### AUTOMOTIVE

Major outlets are: bus and lorry chassis, wheels, primer surfacer, interior trim, steering columns, bumpers, rocker box covers, brake cylinders, seating frames.

#### **Properties**

The requirements will vary with the end use but high corrosion resistance is a prime feature.

Resistance to A.S.T.M. salt spray in excess of 2,500 hr.

100% humidity resistance (without impairment of adhesion and flexibility characteristics) after 2,500 hr.

Scratch resistance in excess of 4000 gm. and resistance to gravelometer and chipping tests.

Gloss requirements may be as low as 15% for primers with high gloss for one coat finishes.

Epoxy based powder coatings are suitable for all the above outlets.

#### ELECTRICAL

Major outlets are: switch gear cabinets, bus bar insulation, coil windings, resistors and printed circuits.

#### Properties

Good insulative properties are required at film thicknesses of around 15 thousandths of an inch (380 microns)

Fluidized bed application (or a special modification), is the most common coating method used to obtain the high film thicknesses.

Epoxy coatings are suitable for most electrical applications.

#### **EXTERIOR QUALITY**

Major outlets are: metal window frames, garden furniture, cycles, street lighting poles, construction metal work, siding enamels.