



# POWDERED AND PARTICULATE RUBBER TECHNOLOGY

**COLIN W. EVANS**

F.P.R.I., F.I.R.I., A.R.T.C.S., F.Inst.Pet.

*Technical Manager, Dunlop Limited,  
Gateshead-on-Tyne, England  
Chairman, Hose Technical Committees,  
British Standards Institution  
and International Standards Organisation*



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To my wife, DOT

## PREFACE

Considerable effort has been spent in the early 1970s in developing the concept of processing powdered rubbers in a manner similar to that used within the plastics industry. Indeed, the large amount of work already published and referred to throughout this book is an indication of the tremendous success already achieved by the polymer manufacturers, by the end users and also by the rubber machinery manufacturers.

A Delphi study† predicted that, by 1980, 25% of the total rubber production will be in crumb or powder form. It was also predicted that, by 1985, powder will account for 20% of the extruded elastomers. In addition, there is a definite trend towards automatic weighing and continuous mixing. Powdered and particulate rubbers lend themselves well to this latter trend. These predictions are perhaps now optimistic by two or three years because of the worldwide trade recession and, to a degree, the reluctance within the industry to adopt this new approach to rubber mixing and processing. This reluctance has been based upon technical, economic and practical concerns, and also to apathy or stubbornness in some areas.

The important point always to consider is that of keeping an open mind, and at least to review existing plant, equipment and processes critically, to see whether the powder concept has any place in future operations, when new equipment is required.

It is believed that the powdered/particulate principle will continue to make inroads as developments, particularly in the machinery and processing field, continue to be made and as co-operative efforts continue.‡ Obviously, the rubber industry will not change overnight, because of the amount of investment it has in conventional mixing equipment, but as this type of equipment wears out and requires replacement, the continuous mixing of powdered rubber should be

† Delphi Study (1972). *Future Rubber Processing*. E. I. Du Pont de Nemours, Wilmington, Delaware, USA.

‡ Schroeder, H. (1975). 16th Annual Meeting of IISRP.

given serious consideration, together with the improved utilisation and efficiency of existing machinery.

It is therefore considered that the future for powdered rubbers is optimistic and bright, and its use will continue to grow.

It is trusted that this book, which is the first to be written on this subject, will serve as a guide and introduction to this very fascinating technology. Much of the work referred to is current practice in the general rubber goods (GRG), electric cable and hose industry fields, but nevertheless the principles can well be applied to other branches of the rubber industry where appropriate.

In the preface to *Hose Technology*, the companion volume to this book, it was mentioned that despite the fact that the product manufacture is a very technical and precise science, it is also very important to consider the reader. The present volume, therefore, in a similar vein, has attempted to provide a combination of authoritative and informative matter with 'readability'. At the end of each chapter are listed references to some of the more useful and recent works on the subject matter, which are intended to indicate authorities and to furnish suggestions for future reading. Many of these publications have been consulted in the preparation of the chapters, and grateful acknowledgement is hereby made.

Also, it cannot be overemphasised that there is no substitute for practical experience, and this too has been taken into consideration for the reader, bearing in mind the difficulty in training, and of keeping lectures, theory and practical work in place. It is one thing to read about riding a bicycle, but quite another matter to do so in practice!

My sincere thanks are tendered to my employers, Dunlop Ltd, and also to my colleagues for reading and checking the manuscript and proofs and also for constructive suggestions.

Special thanks are due to my wife for her usual forbearance, good humour, and encouragement during the months of preparation, and also to my secretary, Mrs Linda Scott, without whose assistance in secretarial work the compilation and preparation would have been extremely difficult or indeed impossible to complete.

I would like also to place on record my thanks to all the various plant manufacturers who have kindly provided photographs and illustrations (separate and individual acknowledgements are made in the text), to the raw material manufacturers who have supplied materials and advice, especially Bayer, BP Chemicals, Cabot Carbon, Goodyear Chemicals and Imperial Chemical Industries Ltd, and also to my many friends and colleagues within ISO, BSI and the British and American Rubber Manufacturers' Associations.

Where machinery manufacturers and suppliers are mentioned by

name, offence is neither intended nor inferred to others not mentioned. It so happens that the stated equipment or material happened to be seen or used, but obviously there are other items, etc., that are also available and can be used.

Finally, where possibly controversial matters are discussed, the views expressed are my own and not necessarily those of my company.

COLIN W. EVANS

*Low Fell*  
*Gateshead*

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## CHAPTER 1

# HISTORICAL

Without doubt, the most efficient and economical method of mixing and compounding elastomers is by the powder route, but before discussing this in detail it is necessary to consider, historically, conventional methods in everyday use within the industry, using 'baled' rubbers.

### MIXING FROM BALED RUBBERS

As a very sweeping statement, one could say that equipment used for mixing the compounds may be broken down into three categories:

- (a) Open mill mixing.
- (b) Internal mixing.†
- (c) Continuous, automatic and/or high speed mixing.

It is obvious that the cooling of the mixing equipment at most stages of the process is of paramount importance, and much development time has been devoted by the various makers of the mixing equipment over the years. This, without doubt, has made the current and faster mixing cycles possible.

For reasons of speed, output and economy, the internal mixing operations are the ones most widely used, but in the hose and GRG fields, open mill mixing is still widely practised, and no doubt will be so for many years to come. This is because of the small production runs of certain products, and also where precision dispersion is vital, together with the need for no contamination. Both of these important features cannot be guaranteed with internal mixers.

#### **Open Mill Mixing**

Very briefly, the open mill mixing process is used to masticate the polymers until an even and smooth band is formed around the front

† Farrel-Bridge Ltd, Castleton, Rochdale, UK (Banbury mixer). Francis Shaw Ltd, Clayton, Manchester, UK (Intermix mixer).

roller. The fillers and oils are added alternately, followed by any small additions and finally the vulcanising materials. During the whole operation, cutting and blending by hand rolling is carried out. As the powders drop into the mill tray, they are swept to the front by the operator and added back into the mill nip. The mill tray is usually slightly sloping to help the operator, and a very useful aid is to fit a vibratory mechanism so that the powder is continuously returned to the operator, thus saving physical effort.

With those compounds containing mixed polymers, the best dispersion and blending of these is obtained by individual breakdown of each polymer, and then blending them together while they are still hot. The addition of the fillers, etc. then follows in the usual way. In order to help breakdown, special peptisers may be added during mastication and, in the case of polychloroprene, retarders and other processing aids may be added very early in the mixing cycle.

### **Internal Mixing**

The desirable set-up for internal mixers is to have two mills in the chain. The first is used to remove the heat from the compound rapidly, prior to the addition of the vulcanising ingredients on the second mill. This also has the advantage of keeping the internal mixer free from curing materials, with far less tendency to give scorch problems. It could be claimed that such a system is expensive from the point of view of initial capital cost of plant, but on balance the ease of processing of the resultant compound during subsequent operations more than justifies this extra cost and many factories are installed in this manner. As with open mill mixing cycles, the general operation is mastication in the mixer and, when the polymer has reached the desired state, the powders, etc. are added, for mixing and dispersion to take place. The batch is now dropped, passed through the first mill several times and then the curatives are added on the second mill, as stated above. It cannot, however, be overemphasised that the exact mixing procedure used, whether open mill or internal mixer, depends on local conditions peculiar to the particular factory, and indeed upon the wishes and experience of the factory compounder. It is very necessary to be able to keep an open mind on the exact technique to be used.

The following techniques, which are widely practised, will illustrate the point.

#### *Direct Mixing Process*

This means exactly what is stated, in the sense that the compound is mixed and is then fed directly to the next operation, i.e. either to an

extruder or calender. This process obviously requires the accurate planning of the mixer cycle and subsequent operations, and has fairly widespread use. It is most necessary to have tight quality control in operation for several reasons, such as curing and dispersion properties, and also the initial mastication is extremely important because of subsequent 'nerviness', as there is no maturing time in the cycle.

#### *Indirect Mixing Process*

In this cycle, the compound is mixed and slabbed off and stored prior to the next operation. The curing materials may be added either before slabbing or just prior to the next operation, after maturing in storage, depending on the particular compound. It is considered that this system (apart from open mill mixing) gives the best bale rubber compound, from a processing angle.

#### *Premastication*

In certain instances, particularly in compounds where there is a fairly high hydrocarbon content, it is necessary to premasticate the polymers (to remove nerve), slab off and cool, and then mix in the normal way with this premasticated material. Alternatively, if the compound contains a fairly high filler content, whether black or mineral, a 'master batch' or 'mother stock' may first of all be mixed, slabbed and cooled, and then finally mixed.

#### *Oil Extension*

In the case of natural rubber compounds it is possible to oil extend, without undue loss of subsequent processing or physical properties, by the selection of a suitable oil and preblending this with the requisite carbon black. The 'carboil' so produced is added to the internal mixer with the rubber right at the beginning of the cycle, and the whole is then masticated together. This technique prevents undue scission due to mastication, and the resultant plasticity so obtained is most satisfactory. The addition of dihydrazine sulphate to the compound also helps subsequent processing of such oil-extended natural rubber compounds.†

#### *Dump Mixing*

When the compound contains a fairly high proportion of filler, it is sometimes difficult to get the rotors of the mixer to 'bite', and in such cases it is normal literally to dump the whole of the ingredients

† Anchor Chemical Company Ltd, Clayton, Manchester, UK.

(rubbers and fillers) into the mixer together, and then carry on with the cycle. Contrary to past beliefs, this technique does work and produces a satisfactory compound for its purpose.

#### *Upside Down Mixing*

This technique consists of first adding the powders to the mixer and then following this with the polymer. This not only produces a satisfactory mixed material, but also makes it possible to mix certain difficult polychloroprene recipes, which hitherto had to be carried out on open mills because of scorch and/or sticking problems.

#### *Seeding*

This is another extremely useful mixing technique, (BICC Ltd, Leigh, Lancs., UK; private communications), which was developed during World War II because of shortages of natural rubber and other hydrocarbons. It consists of the addition of a small portion of the previous batch of the same material, and allowing the new batch to 'seed' on it during mixing. This is particularly effective in mixing extremely low hydrocarbon content compounds.

#### BATCH SIZE

It is extremely important periodically to check the wear on both the rotors and shells of the mixers and to increase the batch weights slightly from time to time to compensate for this, otherwise various mixing difficulties will occur, such as dispersion, lack of mastication, etc. It is surprising just how much more good compound can be obtained by these additions, as extra production, before the machine requires reconditioning.

#### CYCLES

There are two schools of thought on the techniques to be used in the mixing cycle of an internal mixer. One is to mix to a fixed time cycle and ignore the ultimate temperature reached; the other is to mix to a fixed temperature rise on mastication and ignore the total time. Both of these systems produce uniform and economic material from a cost and production angle, but it would be very unwise to say categorically that one was better than the other. In fact, it may even be necessary to operate both systems (in different machines, of course) to suit the particular and individual recipe.

Wherever possible it is now current practice to mix as near as continuously as is practicable. Furthermore, the use of high speed rotors is increasing. This considerably reduces the mixing time but

increases the temperatures. Because of this, as mentioned earlier, a lot of work has been carried out on very efficient water cooling systems and the rapid discharging of the mixed compound. This includes drop doors.

In order further to speed up operations, and by selecting the correct mixing procedure, coupled with accelerator and curing systems, it is possible to direct process mix the whole batch in the mixer. This would not have been possible a comparatively short time ago. Additionally, by mother stocking certain of the more difficult accelerators it is also possible to add these and blend away rapidly without scorch, right at the end of the internal mixing sequence. Very recent technology involves use of the so-called power integrator, which ensures that the mixing is carried out by monitoring and using the electrical energy efficiently and in the correct place, thus ensuring uniform and constant plasticity from batch to batch.

#### SYNTHETIC MATERIALS

Polychloroprenes have been mentioned earlier, but it is considered worth stating that specially prepared magnesium oxide, in 'stick' form, considerably helps dispersion of the magnesium oxide at the critical stage of mixing, with a reduction in mixing time and temperature rise.† Thus, desirable processing properties are conferred to the mix.

Nitriles are fairly straightforward on open mills, but are rather difficult in the usual type of internal mixer because extremely high electrical energy is required. However, by using specially designed rotor blades, even this material can be made to process well.

When butyl rubbers are processed it is desirable to have the mills so positioned as to be able to work safely on the back roll, due to the affinity of this polymer for the faster roll. In larger user factories it is possible to keep butyl rubber confined to its own line, thus making it practicable to adjust the mill gear ratios so that the rubber is banded around the 'front' roller.

#### CONTINUOUS AND SEMI-CONTINUOUS MIXING

In the period before the mid-1960s there had been very little change in the methods or the equipment used within the mixing rooms of the rubber industry. Indeed, the principles and some of the systems used

† Newalls Insulation Ltd, Washington, Durham, UK.



nearly a century ago were still current. It is only perhaps over the past five years that systems anywhere near approaching the ultimate goal of continuous mixing† have been introduced, with varying degrees of success, and also in many cases perhaps at too high a cost when compared with the more established methods. Because of the various polymers used, the mixing equipment must of necessity be very strong and robust to take care of the very high loads and stresses developed during processing.

The future path of continuous mixing could, however, depend very much on the success of the resurrection of the so-called newer technologies of liquid rubber processing (S.M. Morrel, RAPRA, Shawbury, Salop, UK; private communications) and also of powdered rubbers.<sup>1</sup> Over the years these technologies have had limited success, mainly due to deficiency in materials, methods and processing machinery, but currently stand much greater chance than hitherto, and worldwide a considerable amount of work is going on in these fields.

The rubber industry has always been beset by the physical difficulty of mixing and processing rubber, i.e. the forces necessary during the mixing stage, to break down the material before the fillers and other ingredients can be incorporated and blended away. There is a continual search for an easy and cheaper way of achieving this. On more than one occasion latex technology (S. M. Morrel; private communications) has been looked at, as this appears to be a comparatively easy way of getting dispersion and good mixing with the minimum of shear and hence lower power consumption. There has, of course, always been the same stumbling block—the presence of water and its removal. This then led to the use of solvents, with the problems of fire and/or health hazards due to the presence of toxic materials. Finally, rubber was liquefied by melting,‡ thus producing depolymerised rubber, with a viscosity of about 5000 poise at 20°C. In the 1930s, fillers were added to this material on rubber mills and excellent insulation material was thus produced.‡ More recently, low molecular weight SBR materials have been produced, with viscosities of around 500 poise at 25°C.

The present difficulty is still to obtain dispersion, in particular of the carbon black, while at the same time retaining a pourable material. Furthermore, whereas it is possible to disperse in Z-blade mixers, the black in particular remains in large aggregates even after prolonged and lengthy mixing, and the ultimate physical properties, especially flex, are inferior.

† Farrel-Bridge Ltd, Castleton, Rochdale, UK.

‡ BICC Ltd, Leigh, Lancs., UK.