SIGNUS

SISTEMA COLECTIVO DE GESTIÓN DE NEUMÁTICOS FUERA DE USO

Guide for the Production of Rubberised Binders

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Original Work

Guía para la fabricación de betunes con polvo de neumático

EXEMPTION FROM LIABILITY

The contents established in this guide are the analysis results of different authors' experiences previous to this Project and the synthesis of the experimental results carried out with different bitumens and crumb rubber in the laboratory.

The authors of the present study have developed the Guide with the highest technical accuracy and criteria. However, ultimately, it is up to the good judgement of the user to guarantee satisfactory results and therefore the authors and SIGNUS Ecovalor shall not be held liable regarding any derived practical implementation.

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OUTLINE

SIGNUS Ecovalor is a non-profit organisation created as a mechanism for all those tyre manufacturers and importers/producers who wish to comply with the legal obligations related to the end of life tyres management.

The aim of SIGNUS is to guarantee the correct treatment of end of life tyres (ELT), from their generation until they are converted into valuable raw material; maximised through the development of new applications and new markets, one of the most important the asphalt mixtures.

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Despite the existence of a work agreement, SIGNUS as the promotor of the document would like to expressly thank Professor Juan Gallego for his determination and interest regarding this topic. Thanks to his hard work the Project at hand has reached high levels of technological development and innovation with an industrial approach which makes it extremely valuable as a way of boosting the development of rubberised asphalt mixtures in our country.

Similarly, the implication in this Project has allowed us to obtain a considerable amount of outstanding information and hence has required an enormous effort to summarize the contents and thus provide a simple and direct tool for those beneficiaries, public administrations, construction companies, asphalt mixtures manufacturers, engineers, etc. This all leads to a change in perception regarding the use of crumb rubber in asphalt mixtures.

In the same way, SIGNUS appreciates the additional effort made by Professor Juan Gallego concerning the communication of the results obtained in this Project, as well as general technologies by means of his participation in symposiums and seminars for the dissemination of the use of crumb rubber in asphalt mixtures.

Finally, we would like to thank BONDERALIA MONTOIL, IKA, SILVERSON and TECNIDOS SISTEMAS Y PROCESOS for their permission to use images of their equipment in this document.


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Guide Presentation

1

The Technical Specifications for Road Works and Bridges (PG-3) established by the Spanish Ministry of Public Works and Transport, states that it is possible to use binders and asphalt mixtures modified with polymers providing they comply with the established specifications.

In this sense, the modification of bitumen can be carried out using polymers or crumb rubber[1] (Circular Order 21/2007 Ministry of Public Works and Transport) derived from end-of-life tyres. This yields different types of binders and asphalt mixtures with different properties yet always superior compared to those obtained using conventional bitumen.

Apart from the positive effects achieved with crumb rubber, one of the most favourable consequences and thus making worthwhile the generalized use is the fact that with the current price of bitumen it is possible to reach solutions which improve the rheology at a lower cost than when using polymers and other additives.

Together with this initial cost differential, the following advantages must be mentioned: road conservation cost savings and longer service life.

SIGNUS, an organization formed with the primary objective of guaranteeing adequate handling of end of life tyres (ELT) collected in Spain, meets the demands of management as well as fulfilling additional requirements established by the General Administration and the Autonomous Communities through the Waste National Plan (PNIR 2008-2015). This Plan establishes as an objective that 55% of rubber obtained from ELT should be used in asphalt mixtures.

"The use of rubberised asphalt mixtures means savings now and in the future"

(1) Throughout the Guide, the authors use the term ELT powder or crumb rubber in reference to the rubber powder obtained from the transformation process of end of life tyres.

Up until now, there is no official register with specific data regarding ELT powder consumption in Spain nor regarding the road sections paved with asphalt mixtures containing crumb rubber. Nevertheless, approximately 1,100 km of road works are estimated to have been paved with this material. The positive results achieved over the last few years support the confidence placed in this technology.

Although some companies have endeavoured to develop these applications, there is still a fair amount of practical unawareness and fear when it comes to tackling the formulation of rubberised binders. This probably stems from the assumption that a great deal of technical development is required.

The present market analysis regarding rubberized binder manufacturing reveals that only a few producers are commercialising this kind of binder. These companies are highly specialised and carry out their own work formula. It is common that in dissemination documents regarding their products, they describe the properties and improvements obtained without mentioning the formulations used as this remains part of their know-how.

This situation means, among other consequences, that mid-size companies with little experience with bitumens do not venture to produce bitumen even though the equipment required is fairly simple and cost savings are significant.

This guide is aimed at those construction companies or producers of modified binders with limited specialisation and technical requirements with regards to rubberised binder manufacturing. In this sense, we aim to provide a simple document which will allow

these companies a pre-formulation of the most commonly used rubberised binders in Spain. Therefore, with a few preliminary verification tests, they will be ready to manufacture these products.

In order to achieve the objective mentioned above, a series of lab tests have been proposed to determine some approximate composition ranges for rubberised binders to comply with the technical requirements established in the Circular Order 21/2007 of the Spanish Ministry of Public Works and Transport, and the article 212 of PG-3 regarding rubberised binders.

Apart from these pre-formulations, this document includes information regarding the production process (temperature, time, energy agitation, etc.) and store recommendations. Hence the guide user will be able to produce the binders proposed.

"At present an estimated 1,100 km of road works have incorporated this material. The excellent results over the years reinforce the trust placed in this technology"

Regulatory Framework for the Use of Rubberised Binders

2

In Spain, some very important breakthroughs have been made regarding bituminous binder specifications with crumb rubber. Although without a normative character, perhaps the most important milestone before regulations was the document *Manual de Empleo de Caucho de NFU en Mezclas Bituminosas 2007* (*Manual for the Use of ELT Crumb Rubber in Asphalt Mixtures*), drafted by CEDEX on behalf of the Ministry of Environment and the Ministry of Public Works and Transport. This document outlines the three types of rubberised binders that were considered interesting in Spain within the asphalt mixture industry.

In this document, a definition was listed by order of modification, rubber enhanced, rubber modified and high viscosity rubber modified binders, all with crumb rubber derived from ELT.

Several months later the Circular Order 21/2007 of the Ministry of Public Works and Transport was published on the use and specifications to be fulfilled by binders and asphalt mixtures containing ELT crumb rubber, becoming the main document concerning production and use of binders with crumb rubber in Spain.

In the foreword of the Circular Order, crumb rubber is defined as a product obtained from the ELT grinding down to sizes below 1 mm and containing no more than 15% of fine particles smaller than 0.063 mm. It is also specified that it must be composed mainly of natural and synthetic rubbers and maximum percentages of ferromagnetic, textile or other impurities below, 0.5% y 0.25% by weight respectively.

Furthermore, two manufacturing processes are distinguished. The first process is called "terminal blending" and refers to traditional installations for bitumen modification, equipped with a shear mixer, which provides sufficient stability to the binder in order to be transported to an asphalt mixture production plant.

The second procedure is called "production in situ" and consists of a special unit for the manufacture of rubberised binders within the asphalt mixture production plant and refers to units that do not necessarily dispose of a shear mixer.

As for the types of rubberised binders, the Circular Order 21/2007 distinguishes three categories based on the degree of modification:

- Rubber enhanced binders
- Rubber modified binders
- High viscosity rubber modified binders

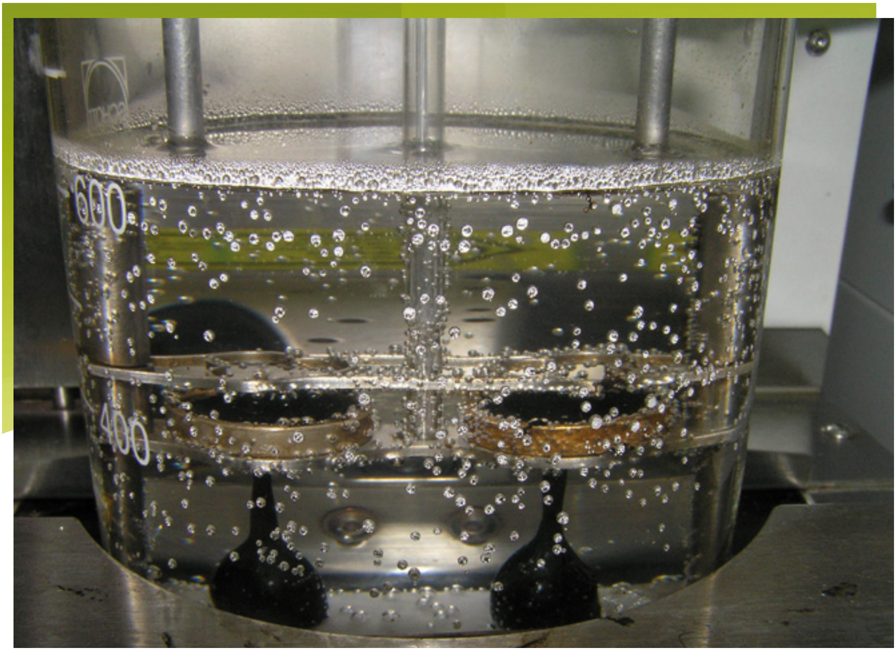
The specifications that are required for each one of the three types of binder coincide or at least are related to the requirements of the modified binders with conventional polymers, set out in the regulation EN 14023:2010.

It is necessary to clarify that the peculiar nature of rubberised binders implies that some of the standardised tests for polymer modified binders (PMB) are difficult to carry out at a practical level. The reason being that there are particles in suspension as the interaction with the bitumen does not mean the total integration of the rubber in the liquid phase. In particular, the cohesion test known as "Force Ductility" (EN 13589 and EN 13703) requires that the bitumen samples reach an elongation of at least 40 cm. However, the heterogeneity caused by the rubber particles within the bitumen prevents these elongations before breaking, even at 25ºC.

In this sense, it is necessary to point out that the regulation EN 14023 regarding polymer modified binders indicates that the cohesion property can be measured with other tests like the Vialit pendulum impact (EN 13588) which seems to be the most suitable for rubberised binders.

Something similar takes place during the elastic recovery test (EN 13398), where a minimum elongation of 20 cm is required and is not usually reached. However, the standard allows, in this case, the possibility of reaching the elastic recovery percentage considering the elongation reference reached at the breaking point and not the 20 cm established in the standard procedure.

In any event, there are no doubts regarding the enhanced properties of rubberised binders, both in terms of cohesion or elastic recovery. This is merely due to an inadequate test procedure taken as a reference at the time of writing the specifications for these rubberised binders.



2.1 Rubber enhanced binders

The characteristics of these rubber enhanced binders were not collected in any legislation and hence, the Circular Order 21/2007 establishes particular specifications for them. In order to indicate the content of crumb rubber, the abbreviation "BC" is followed by the penetration value of the binder. The technical specifications are shown in the following table:

Characteristics		Reference Standard	Unit	BC35/50	BC50/70
Needle penetration at 25ºC		EN 1426	0.1 mm	35-50	50-70
Softening point - Ring and Ball method		EN 1427	ºC	≥ 58	≥ 53
Fragility point Fraass		EN 12593	ºC	≤ -5	≤ -8
Force-ductility (5 cm/min)	5ºC	EN 13589 EN 13703	J/cm²	≥ 0.5	
Elastic recovery at 25ºC		EN 13398	%	≥ 10	
Storage stability (*)	Softening point difference	EN 13399	ºC	≤ 10	
	Penetration difference		0.1 mm	≤ 8	≤ 10
Solubility		EN 12592	%	≥ 92	
Flash point open cup		EN ISO 2592	ºC	≥ 235	
Durability – Resistence to RTFOT EN 12607-1					
Mass variation		EN 12607-1	%	≤ 1.0	
Retained penetration		EN 1426	% o.p.	≥ 65	≥ 60
Softening point variation		EN 1427	ºC	min -4 max +8	min -5 max +10

(*) Only required by binders that are not produced "in situ"

Table 1. Spanish specifications for rubber enhanced binders (BC)

2.2 Rubber modified binders

These are binders that, according to the Circular Order 21/2007, have been modified with crumb rubber (the rubber content is not prescribed, neither whether the modification is achieved with other additives). These binders must comply with specifications of modified binders (article 212 PG-3). They are named adding the letter "C" at the end of the nomination of the corresponding modified binder. The specifications are summarised in the following table:

Characteristics		Reference Standard	Unit	PMB 10/ 40-70 C	PMB 25/ 55-65 C	PMB 45/ 80-60 C	PMB 45/ 80-65 C	PMB 45/ 80-75 C	PMB 75/ 130-60 C
Needle penetration at 25°C		EN 1426	0.1 mm	10-40	25-55	45-80	45-80	45-80	75-130
Softening point - Ring and ball method		EN 1427	°C	≥ 70	≥ 65	≥ 60	≥ 65	≥ 75	≥ 60
Force-ductility (5 cm/min)		EN 13589 EN 13703	J/cm²	≥ 2 at 15°C	≥ 2 at 10°C	≥ 2 at 5°C	≥ 3 at 5°C	≥ 3 at 5°C	≥ 1 at 5°C
Fragility point Fraass		EN 12593	°C	≤ -5	≤ -7	≤ -12	≤ -15	≤ -15	≤ -15
Elastic recovery at 25°C		EN 13398	%	TBR	≥ 50	≥ 50	≥ 70	≥ 80	≥ 60
Storage stability (*)	Softening point difference	EN 13399 EN 1427	°C	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5
	Penetration difference	EN 13399 EN 1426	0.1 mm	≤ 9	≤ 9	≤ 9	≤ 9	≤ 13	≤ 13
Flash point open cup		EN ISO 2592	°C	≥ 235	≥ 235	≥ 235	≥ 235	≥ 235	≥ 220
Durability - Resistence to RTFOT EN 12607-1									
Mass variation		EN 12607-1	%	≤ 0.8	≤ 0.8	≤ 1.0	≤ 1.0	≤ 1.0	≤ 1.0
Retained penetration		EN 1426	%	≥ 60	≥ 60	≥ 60	≥ 60	≥ 60	≥ 60
Softening point increase		EN 1427	°C	≤ 8	≤ 8	≤ 10	≤ 10	≤ 10	≤ 10
Softening point decrease		EN 1427	°C	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5

(*) exclusively for binders not produced "in situ"

TBR: To be reported

Table 2. Spanish specifications for rubber modified binders

2.3 High viscosity rubber modified binders

These binders have certain specifications considering the relevance of the viscosity value to be reached. These are named with the initials BMAVC and the specifications are as follows:

<table>
<tr><th colspan="2">Characteristics</th><th>Reference Standard</th><th>Unit</th><th>BMAVC-1</th><th>BMAVC-2</th><th>BMAVC-3</th></tr>
<tr><td colspan="2">Needle penetration at 25°C</td><td>EN 1426</td><td>0.1 mm</td><td>15-30</td><td>35-50</td><td>55-70</td></tr>
<tr><td colspan="2">Softening point - Ring and Ball method</td><td>EN 1427</td><td>°C</td><td>≥ 75</td><td>≥ 70</td><td>≥ 70</td></tr>
<tr><td colspan="2">Fraass breaking point</td><td>EN 12593</td><td>°C</td><td>≤ -4</td><td>≤ -8</td><td>≤ -15</td></tr>
<tr><td rowspan="2">Force-ductility
(5 cm/min)</td><td>5°C</td><td rowspan="2">EN 13589
EN 13703</td><td rowspan="2">J/cm²</td><td>-</td><td>≥ 2</td><td>≥ 3</td></tr>
<tr><td>10°C</td><td>≥ 2</td><td>-</td><td>-</td></tr>
<tr><td colspan="2">Consistency (Floater, 60°C)</td><td>NLT 183</td><td>s</td><td colspan="3">≥ 3000</td></tr>
<tr><td rowspan="2">Dynamic viscosity</td><td>135°C</td><td rowspan="2">EN 13302</td><td rowspan="2">mPa·s</td><td></td><td>≤ 7500</td><td>≤ 5000</td></tr>
<tr><td>170°C</td><td>≥ 2000</td><td>≥ 1200</td><td>≥ 800</td></tr>
<tr><td>Elastic recovery</td><td>25°C</td><td>EN 13398</td><td>%</td><td>≥ 10</td><td>≥ 20</td><td>≥ 30</td></tr>
<tr><td rowspan="2">Storage stability (*)</td><td>Softening point difference</td><td rowspan="2">EN 13399</td><td>°C</td><td colspan="3">≤ 5</td></tr>
<tr><td>Penetration difference</td><td>0.1 mm</td><td colspan="3">≤ 20</td></tr>
<tr><td colspan="2">Flash point open cup</td><td>ISO 2592</td><td>°C</td><td colspan="3">≥ 235</td></tr>
<tr><td colspan="7">Durability - Resistence to RTFOT EN 12607-1</td></tr>
<tr><td colspan="2">Mass variation</td><td>EN 12607-1</td><td>%</td><td>≤ 0.8</td><td>≤ 0.8</td><td>≤ 1.0</td></tr>
<tr><td colspan="2">Retained penetration</td><td>EN 1426</td><td>% o. p.</td><td colspan="3">≥ 60</td></tr>
<tr><td colspan="2">Softening point variation</td><td>EN 1427</td><td>°C</td><td colspan="2">min -4 max +10</td><td>min-5 max +12</td></tr>
</table>

(*) exclusively for binders not produced "in situ"

Table 3. Spanish specifications for high viscosity rubber modified binders (BMAVC)

2.4 Storage conditions of rubberised binders

Following the Circular Order 21/bis 2009 of the Spanish Ministry of Public Works and Transport, new requirements were included regarding these bitumens; particularly regarding storage and handling of the bitumen at the worksite. It states that all binders with rubber regardless of category and production process should be stored in a tank during at least 8 hours and for a maximum of 72 horas, before being used in the production of bituminous mixtures. The tank must be, according to the Circular Order 21bis/2009, preferably vertical and have a heating system, agitation and recirculation as well as collection points for samples at different levels of the vertical column.

It is important to point out that, storage periods over 72 hours can have a negative effect on the properties of the product. However, according to the recognised Spanish experience certified by excellent practical results, interaction time (production + storage) of 1 hour is necessary to reach digestion and comply with binder specifications, even in the case of high viscosity rubber modified binders.

Interaction Bitumen-Rubber: Digestion Process

3

The interaction at high temperatures of the bitumen with ELT rubber particles has been outlined in different ways in the bibliography. On the one hand, it is described as a chemical reaction that consists of the degradation of the elastomeric chains due to the thermal effect and on the other hand, a process of devulcanization of the rubber; destruction of sulphur-sulphur bonds which is characteristic of the vulcanization brought to light by the decrease of sulphur on the surface of the rubber particles. However, the interaction is mainly a physical phenomenon whose principal mechanism consists in the swelling of the rubber as it captures the lighter fractions of the bitumen.

3.1 The digestion process of rubber in bitumen

During the interaction between rubber and bitumen, the rubber particles start swelling, increasing their volume due to the absorption of aromatic fractions ***(Figure 1)***. Surrounding the rubber particle, a "gel" or "soft layer" forms which detaches as a result of the agitation as it integrates with the bitumen. Taken to an extreme, if the temperature or the agitation energy is prolonged the rubber particle would disappear inside of the bitumen.

The interaction of rubber with the bitumen is called **digestion**.

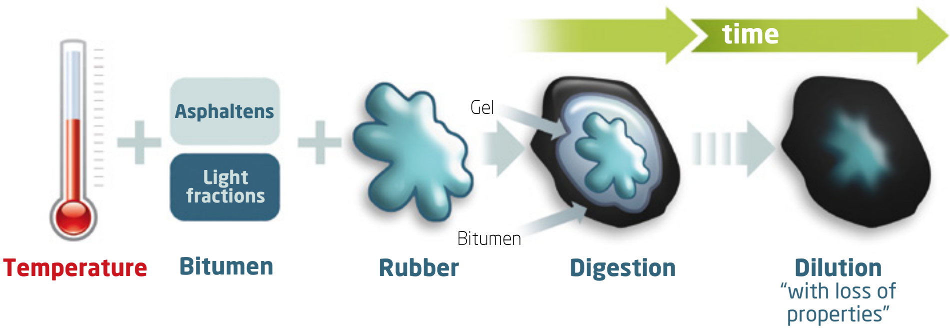


Figure 1. Evolution of the interaction bitumen-rubber

Figure 2 shows a diagram of the evolution of the digestion process over time with the adequate digestion temperature and mixing energy combination that ensures the integration of the rubber improving the properties of the bitumen. However, when the process is prolonged excessively, the improvement is reduced due to the dilution of the rubber in the bitumen.

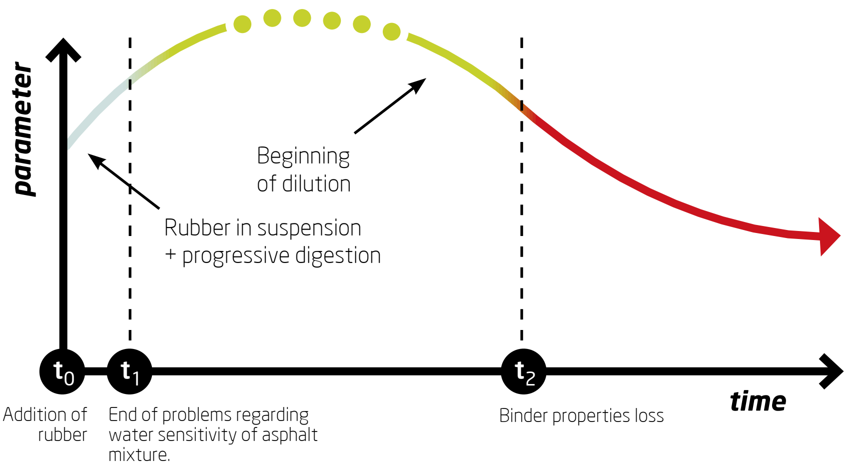


Figure 2. Evolution of modified properties

There are several techniques used to study the digestion process of the rubber in bitumen. Among others, filtered rubber in suspension, scanning electron microscopy (SEM), bitumen fractioning (SARA), infrared spectrometry, filtration chromatography in gel and fluorescence microscopy. All these have contributed to an improved understanding of the interaction between rubber and bitumen.

In short, the process of digestion is enhanced:

- As the temperature increases
- When contact time is prolonged
- As the maximum rubber particle size decreases
- The higher the content of light fractions in the bitumen
- As the mixing energy is increased

"An adequate combination of digestion temperature and mixing energy ensures the integration of rubber in the bitumen improving properties"

Characterisation of Crumb Rubber

4

An important aspect when formulating rubberised binders is to know the characteristics offered by the ELT crumb rubber. Different studies show that the composition of ELT crumb rubber and its particle size distribution are two of the main characteristics of the rubber particles that significantly affect the properties of rubberised binders.

The Circular Order 21/2007 defines crumb rubber obtained from ELT as ground rubber from ELT of sizes under 1 mm as long as the particle content below 0.063 mm is less than 15%. Furthermore, this document states that the crumb rubber must be made up of mainly natural rubber and synthetic rubber and must not contain ferromagnetic material, textile or contaminants above 0.01%, 0.5% y 0.25% by weight respectively.

In this chapter, the principal characteristics of ELT crumb rubber are enumerated as well as a description of the testing procedures regarding characterisation.

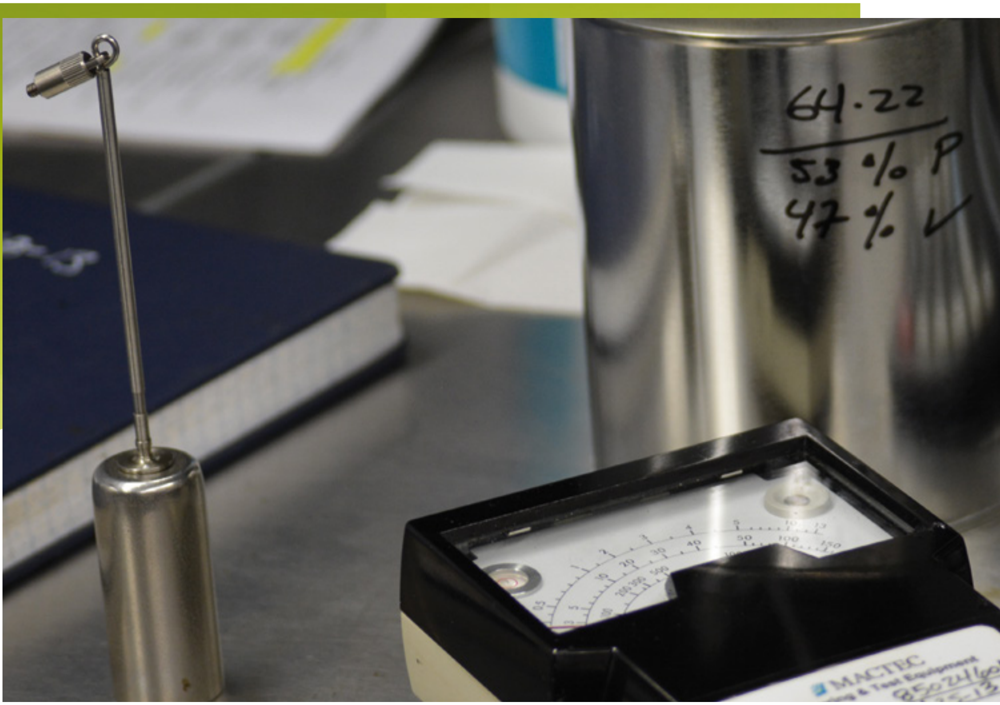
4.1 Characteristics and testing procedures

The principal characteristics of crumb rubber are:

- **Chemical composition:** ELT crumb rubber is made up of a mixture of different types of rubber, mainly natural rubber (NR) and synthetic rubber like SBR and BR. The proportions depend on the origin of the tyre. In this sense, the content of natural rubber from truck tyres is higher than those used on light vehicles (passenger car, van or motorbike tyres).

  For measurement of the content of natural rubber, the only precise method is based on the ^{14}C techniques which determine the biomass content of a sample and which is equivalent in terms of natural rubber content.

- **Bulk Density:** This may vary slightly depending on the production process. Nevertheless, as a reference value an apparent density of approximately 0.5 kg/dm^3 may be used, with



slight oscillations depending on the particle size distribution and the origin of tyre.

- **Particle size distribution** Apart from ensuring that the crumb rubber particles are smaller than a determined size, it is also fundamental to know the size distribution over the grading curve. The test method used to establish the particle size distribution is contained in the European standard CEN/TS 14243: 2010 of experimental character and entitled: "*Materials produced from end of life tyres- Specification of categories based on their dimension(s) and impurities and methods for determining their dimension(s) and impurities.*"

  The test method used in order to determine the particle size distribution consists of introducing a sample of at least 150 g in a sieving equipment with a vibration movement of at least two dimensions. In this way, the material will be classified by size by means of different sieves. Sieving time will be 10 minutes. The results report provides the particle size distribution curve which includes upper and lower limits as well as the percentages of material that passes through the intermediate sieves placed in the unit.

- **Steel or ferromagnetic material content:** By means of an analysis the quantity of free steel within the crumb rubber is determined. The method entails the use of a magnet in order to extract the steel. The test procedure established in the standard CEN/TS 14243:2010 previously

- **Impurity content:** The experimental standard mentioned before, describes a test method based on densimetry method. This test is carried out by placing a sample of 150 g of crumb rubber on a saline solution of 300 g common salt mixed with 1 litre of water resulting in an approximate density of 1.25 g/cm^3, higher than the density of rubber (around 1.125 g/cm^3). As a result, the foreign objects found in the crumb rubber, such as sand, glass or non-magnetic metal particles, settle on the bottom of the container used.

- **Moisture content:** The analysis will be carried out according to standard UNE 103-300-3 (except for the temperature of the heater, which will be 105±5°C and the minimum sample mass will be 100 g). In this standard, the test method to determine the soil moisture using a drying oven is described.

mentioned proposes the collection of a 500 grams of crumb rubber sample spread out on a tray that allows a maximum thickness of 2 cm. Then, a magnet with a surface area larger than 2 cm^2 and a minimum intensity of 1 Tesla is passed to which the steel particles become attached.

- **Textile content:** To carry out the quantification of this parameter, the standard CEN/ TS 14243:2010 establishes a similar procedure used in the particle size distribution test.

  The same test is carried out during 10 min. The textile fibers, if present, tend to form small ball aglomerations which are then separated and weighed for quantification.

"The test methods used to establish the particle size distribution are compiled in the European standard CEN/TS 14243:2010"

Design of Rubberised Binders

5

The design of rubberised binders is a process comprising the selection of components and manufacturing process in order to achieve thereupon an appropriate reproduction of the process on an industrial scale and the obtention of a product in accordance with the set specifications.

Although the procedure is not excessively complex, the instructions included in this Guide and years of experience will be useful and reduce efforts.

In the following sections, a detailed description, step by step, of the procedure in order to complete the design is described.

5.1 Selection of base bitumen

As a starting point, the base bitumen must be softer than the final sought rubberised binder. The rubber, as well as the additives and polymers used, tend to make the bitumen more consistent at intermediate and high service temperatures. In terms of the Spanish standards regarding binders, the addition of rubber to a base bitumen results in a reduction in penetration and a higher softening temperature.

The chemical nature of the bitumen exerts a certain influence. Generally speaking, the preferred bitumens are those with the highest possible content of naphthene-aromatic and aromatic-polar compounds in accordance with standard NLT-373 (fractionating column). Nevertheless, the influence of the bitumen composition, within the ranges that are frequently administered in Spain, is far less than the penetration influence of the original binder.

5.2 Crumb rubber selection

The most influential parameter regarding bitumen modification is crumb rubber particle size distribution. In general, the coarse grading slows down the bitumen-rubber interaction and accentuates the sedimentation problems of the final product. Hence, continuous grading is advised with maximum sizes of 0.8 mm.

The chemical composition of the rubber also influences the final characteristics of the bitumen. The control parameter should be the percentage of the elastomeric fraction just as the content of natural rubber. In both cases, the highest possible values are preferable.

Finally, the rubber milling method should also be considered: ambient temperature or cryogenic. Generally speaking, it is advisable to mill the rubber at ambient temperature as this provides more specific surface and furthermore it is much more common in the market.

> "The addition of rubber to the bitumen results in lower penetration and higher softening temperature"

5.3 Polymer selection

In the case of modified binders, it is commonplace to use a polymer in order to complete the degree of modification obtained with rubber. Regarding the polymer used, in section 7.1.3 of this Guide, specific indications are provided about the selection. The most efficient polymers to complete the effect of the rubber are those based on elastomers SBS type (styrene-butadiene-styrene). This usually comes in the form of powder, pellets, etc. The choice for using one type or another will depend on the type of mixer used and is explained in the following section.

On the market, there are diverse SBS polymers, with important differences regarding structure, properties and above all the influence that these have on the bitumen when added.

After analysing the properties of the polymers used for the work and the results on the bitumen properties, we conclude that the SBS polymers radial type or branched have a more significant modifying effect on the bitumen. This greater modifying effect compared to lineal polymers is due to the branched structure which facilitates the creation of a three-dimensional network within the bitumen.

5.4 Selection of laboratory mixer

At a laboratory level, there are two types of mixers depending on the rotation speed, high or low speed; and whether the devices develop a shear effect or not.

According to the existing installations at the production plants, it is commonplace to use only mixers when the modification of a binder is carried out with ELT rubber. However, when polymers are used additionally it is appropriate to use an industrial unit that includes a shearing effect.

Therefore, if polymers are incorporated in the laboratory, it is recommendable to use units equipped with shears. If this is not the case the polymer used must be added in powder form in order to facilitate the distribution within the binder.

The type of mixer used in the laboratory should, therefore, align with the mixer available at in-

dustrial level. As an example, if the industrial manufacturing equipment uses a larger mixer and digestion tank, it will be convenient to have an energic first phase mix in the laboratory. This should be followed by a digestion period of continuous agitation of the rubberised binder as occurs in the digestion tank at an industrial level.

Annex 1 of this guide offers some characteristic models of both types.

5.5 Mixing temperature and digestion time

It has been proved that the intensity with which the bitumen and rubber interact, during production, is approximately doubled for each 10°C of temperature increase. Therefore, the higher the temperature the less digestion time required.

Nevertheless, the binders age as they are heated and thus, unnecessary high temperatures should be avoided. Therefore, according to the experience, the mixing temperatures recommended should remain within the following ranges:

- Binders with a rubber content of up to 22%: 185-195°C
- Binders with a rubber content of up to 15%: 175-185°C

It is important to note that although the base bitumen is conditioned at a specific temperature, once the rubber and eventually the polymer are added the temperature will drop in the mixer by 10 to 40°C approximately. This values will depend on the percentage of rubber added and the room temperature. In this sense, following a first mix by hand with a spatula, it is necessary to condition the rubber-bitumen blend at the desired temperature before proceeding.

Regarding digestion time, generally speaking a 60-minute period is required, nevertheless, at an industrial level, this period may be shorter.

5.6 Sample collection and control tests

This aspect is essential. In general, rubberised binder tends to sediment and therefore, the following precautions should be taken when collecting samples:

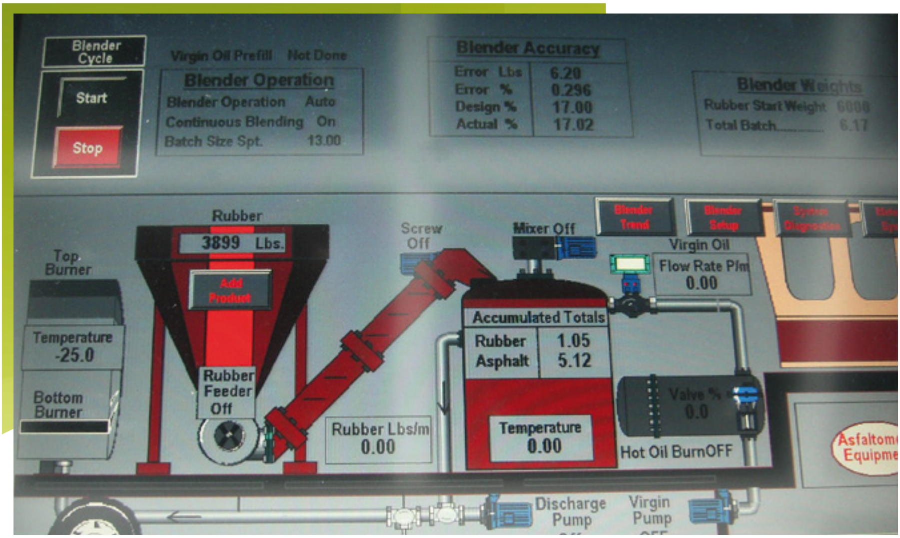
- Stir with the spatula before and during the sample collection.
- Collect the samples in small containers and cool them as quickly as possible.
- If tests are to be made with the same sample over several days, make sure the sample is divided into various portions. In this way, heating can be done individually and the excess material can be thrown away.

As a guideline to carry out frequent tests, a sample size of 650 grams will be sufficient. This quantity may be distributed and used during the different tests.

5.7 Formulation of rubberised binders in the laboratory

Once the specifications have been accomplished after one or two tests, a formulation document will be drafted in the laboratory and must feature the following elements:

- The weight composition of the product: weight percentages of the bitumen, rubber and finally the polymer, if included.
- The specifications of the base bitumen , in particular, the penetration and the origin.
- The specifications of the rubber, in particular, the particle size distribution and the origin of the tyres (in percentages)
- Identification of the polymer, if included. Attention will be given to its commercial denomination as well as its form of presentation: powder, granulates etc.
- The mixing temperature, as well as the digestion temperature when different from the first.



- Minimum digestion time; that is the total time in the mixer and in the digester if available and if different to the first.
- Final product characteristics: Needle penetration, softening point. In the case of high viscosity rubber modified binders, the reference value of the viscosity parameter should also be reported.

This document will serve for the formulation at industrial level.

5.8 Verification of the binder characteristics at industrial level

It is usual to find differences between binders produced in the laboratory and binders produced at industrial level. This is due to the following reasons: the type and energy of the mixer, different exposure to oxygen and therefore different ageing in each case.

The usual verification procedure should begin with the formulation of the binder in the laboratory and continue with the first batch at industrial level while verifying the correspondence with the properties measured in the lab samples. The necessary adjustments should be carried out in order to match the results on a real scale. This is an iterative process which becomes brief and even unnecessary once a certain amount of experience is gained.

Manufacturing of Rubberised Binders at Industrial Scale

6

Once the formulation of the rubberised binder at the laboratory scale is achieved, the next step is production at industrial scale. To this end, there are a series of appliances or equipment whose key principals will be explained as follows.

In this chapter, we also present the different phases of the industrial manufacturing process: the reception of bitumen, rubber and eventually the polymer, binder manufacturing with rubber, production control and final product as well as the storage conditions.

6.1 Systems for the manufacturing of rubberised binders at industrial scale

The production of rubberised binders requires the intimate interaction between the bitumen, the rubber and occasionally polymers as well as other additives that help the bitumen obtain and maintain specific properties.

> ***"The aim of the manufacturing unit for rubberised binders is to facilitate the interaction between components in the shortest possible time"***

6.1.1 Configuration of the most frequent production units

There are two production schemes: in-line mixing also known as continuous and batch mixing.

- **In-line mixing**

During **in-line mixing** the components which are to be mixed pass through the equipment in a continuous manner and leave it completely mixed. This may be observed in ***figure 3.***

Figure 3. In-line mixer

In this type of mixer, the production is carried out without recirculation *(figure 4)* or using a recirculation circuit *(figure 5)* repeating the runs or passes that are necessary for the mixer to reach the degree of integration that will guarantee compliance with the product specifications.

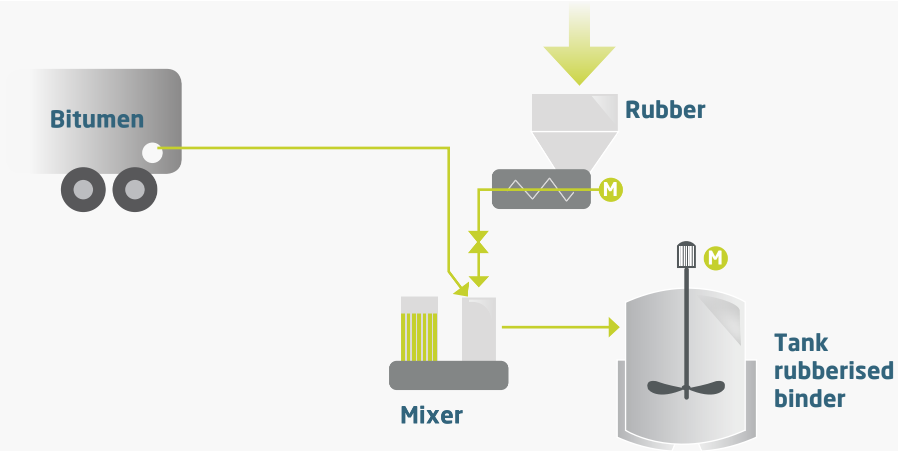


Figure 4. In-line mixer, without recirculation

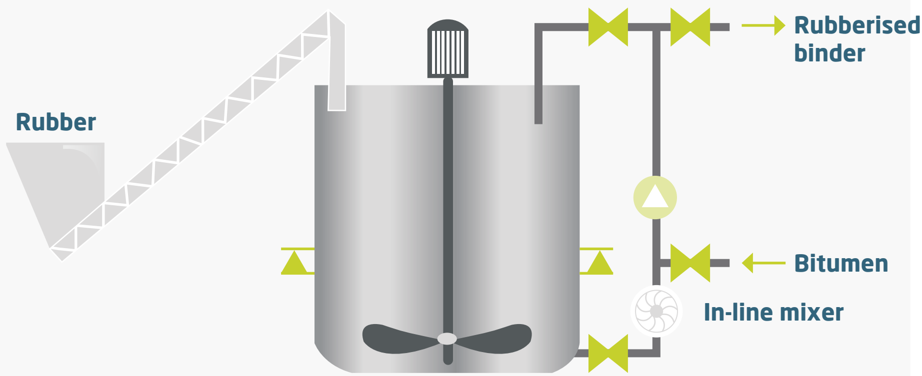


Figure 5. In-line mixer, with recirculation

- **Batch mixer**

Conversely, the scheme used for batch production consists of introducing an agitation device into a tank; in this way, during a pre-set time the rubberised mixture and the eventual additives are in constant movement which enables the precise interaction.

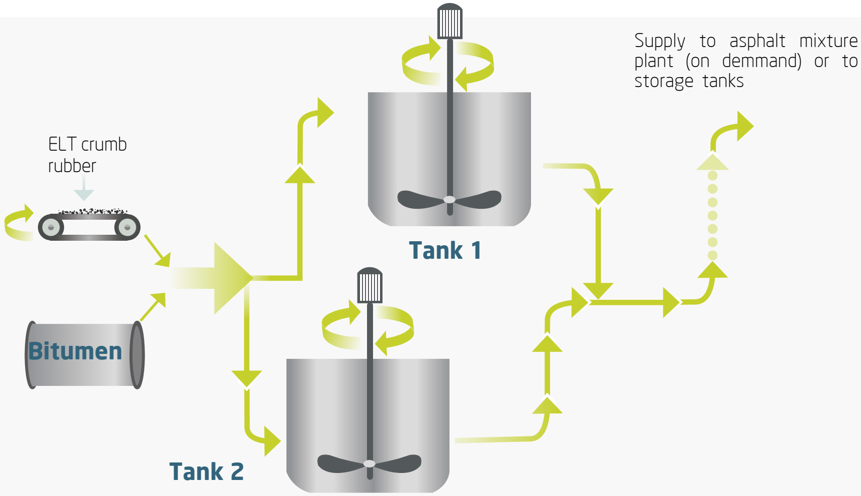


Figure 6. Batch production unit for rubberised binders with two twin tanks

In any case, in order to achieve optimal interaction between the bitumen and the crumb rubber, the configuration of the unit of rubberised binders should be conceived to allow the product to digest the time established in the laboratory. This is calculated by adding the mixing time and the storage time in the digestion tank if available and if different to the mixer.

6.1.2 Types of mixers

Regardless of the configuration of the production unit for rubberised binders, the mixer itself may dispose of a shearing effect or not.

In the first case, the mechanical element is capable of cutting and breaking the particles reducing their size and thus creating new surfaces which facilitate the interaction. This type of mixer usually has a fixed piece, stator, and moving part, rotor, with some clearance between them. By means of suction, the mixture is forced to pass between both pieces producing an intense cutting effect that reduces the particle size of the rubber and increases its specific surface. All

> **"The mixer with shear effect is capable of cutting the ELT crumb rubber particles which promotes the interaction with the bitumen"**

of this promotes and favours the interaction between rubber particles and bitumen.

The traditional mixers without the shearing effect consist of a stirring system that can be more or less powerful which keeps the fluid in movement, thus improving the interaction between the rubber and the bitumen. Without these systems, the interaction would be much slower.

Apart from the two mixers mentioned before, we must place high speed devices (>3,000 rpm) equipped with a head which not only creates a vortex but also cuts and breaks the particles partially.

The decision regarding the choice of configuration and the type of mixer to be used will depend on the manufacturer. In Annex II detailed information concerning the most frequently used equipment within the market has been included: advantages and inconveniences, necessary auxiliary machinery, connection with the bitumen mixing plant, etc.

As a general recommendation, in the case of the use of polymers together with the rubber to obtain certain degrees of binder modification, mixers that use the shearing effect are much more efficient and beneficial.

6.2. Components of the binder formulation

As a starting point the formulation obtained at laboratory level should be used and once the required verifications have been carried out at industrial scale, as indicated in points 5.7 and 5.8. Special attention should be paid to:

6.2.1 Crumb rubber: reception, storage and control

Crumb rubber is usually delivered in tanks as well as in big-bags, with an approximate volume of 2 m^3. On arrival, the specification sheet that accompanies the delivery should be checked. The following data should be indicated:

- Company name and date of production.
- Particle size distribution.
- Proportion, as a percentage, of the origin of tyres used for manufacture.
- Moisture content (%).
- Textile fibre content (%).
- Ferro-magnetic particle content (%).
- Other impurities content (%).

Depending on the quality management system of the material, compliance with quality parameters will be verified, and then the material may be adequately stored (particularly against rain) until consumption. If the big-bags are not prepared with an interior laminate of polyethylene that protects the rubber from rain, they should be covered with a tarpaulin or canvas to protect the contents from the water. If this is not possible, the rubber should be placed under cover.

In order to understand the importance of preserving the stored rubber from moisture, although moisture is lost during the production of the rubberised binder, foaming could take place when humidity and bitumen come into contact. This means a higher risk of splashing or spilling and therefore a lower production rate as a preventive measure.

6.2.2 Polymer: reception, storage and control

Regardless of the presentation of the polymer, this must be accompanied by the corresponding certificate provided by the supplier. Before use, it should be verified that the product corresponds to the type used during formulation in the laboratory.

Regarding the storage conditions; the same precautions should be taken as with the rubber, especially concerning moisture.

6.3 Rubberised binder production

Before commencing production, all the elements of the equipment should be checked to ensure that everything is in perfect working state: heaters, thermal circuit, dosing devices, pumps, etc.

Once production has begun, the temperatures of the base bitumen that enters the production unit and the mixer as well as the temperature of the rubberised binder in the digestion tank, if different from the mixer, should correspond to the production conditions prescribed in the laboratory formulation. In this regard, the unit should be equipped with a system to control and measure the temperatures. These devices should be placed to ensure easy access.

Similarly, the temperature of the tubes, pipe elbows, pumps and other elements that come into contact with the bitumen must be monitored, especially if these are not heated. For this purpose, a thermographic camera may be of great use. The question is to avoid cold blockages within the circuit that the bitumen has to cover and in particular the rubberised binder.

Throughout the manufacturing process, the temperature of the bitumen must be checked sequentially. Necessary actions should be carried out in order to correct deviations from the recommendations established in the laboratory formulation.

Once manufacturing has begun, special attention will be paid to the proportion of components prescribed in the laboratory formulation.

In the same sense, and in order to respect the minimum digestion periods, exceeding the production timetable should be avoided according to design.

6.4 Production control

The viscosity of the binder increases due to the rubber in the bitumen. Furthermore, this property can be measured continuously, in a recirculation tube for example. For this reason, this parameter is used as quality control in real time without the necessity to extract samples. Therefore, it is possible to detect important abnormalities or defects immediately during the manufacturing process.

However, the extraction of samples, together with the temperature control and the manufacture time (including digestion), constitutes the principal element of binder quality control.

To this end, the manufacturing unit should be equipped with the corresponding sample taking valves. So that the sample collected is representative regarding composition, degree of digestion and temperature of the supplied material to the asphalt mixtures production plant, the samples must be collected near the tank outlet.

For an optimum specification control of the bitumen, it is important to discard the necessary amount of binder to ensure that the sample is obtained from the tank and not from the tubes or the sample valve outlet (minimum 3-4 kg).

Likewise, the sample must be made up of another 3-4 kg of rubberised binder, collected in a metallic container of approximately 5 kg in capacity, equipped with a seal to ensure the conservation of the sample.

This sample amount allows, with adequate temperature control, the execution of the viscosity test using a manual viscosimeter. Because of the sample size, the central part maintains the manufacturing temperature during a more extended period of time than if the samples were smaller in size as

is the case of samples from other types of bitumen. The collecting of sub-samples from the field samples should be carried out with the precautions mentioned previously in section 5.6 of this guide regarding sample collection in laboratory.

During the first tests at industrial scale drawn from the laboratory formulation, samples must be collected in order to confirm that the manufactured product complies with the reference specifications presented in chapter 2. The limitations are fully described in this chapter of the Guide.

In terms of frequency, it is recommended that 3-4 kg sample controls should be carried out for every 50 tonnes of product and at least twice a working day. During these controls viscosity *in situ* should be measured using a manual viscometer and from this sample, bitumen sub-samples should be tested in the laboratory to determine the penetration value, the softening point and the elastic recovery. The container with the excess sample remnant should be sealed and labelled, indicating the origin, date and time of day of the sample as well as any possible incidents such as prolonged storage or reheatings. The correctly identified sample will then be preserved for posterior tests if required.

Furthermore, during the manufacture of high viscosity rubber modified binders, a sample collection should be carried out every hour in order to conduct a viscosity test using a manual rotational viscometer. This is also recommended for other types of binders with rubber. All the information obtained concerning viscosity should be compiled in a report and sent to the control organisation or agency.

6.5 Storage of rubberised binders

Generally speaking, the bitumens with crumb rubber are products that should be put to use as soon as possible. The motives for this recommendation may be summarised as follows. In the first place, there is a tendency of particle settling. Secondly, when polymers are incorporated a cream can appear on the surface and finally, the evolution of the properties as the storage time goes by.

Nevertheless, due to the dynamics of the work on site, frequently it will be necessary to store the binders during some hours. The production of rubberised binder and the usage at the worksite is not always synchronized due to the numerous contingencies that arise during road works.

Consequently, it is necessary to find some sort of solution between both realities, the convenience of consuming the product immediately following manufacture and the necessity to operate with a specific storage capacity and regulation in plant provisions.

The following indications are established in order to allow successful storage:

- The storage tanks, in addition to being equipped with heating systems as well as thermal insulation, as usual must be equipped with a stirring system in order to keep the product in continuous movement and avoid settlements or heterogeneities.
- Maximum storage should be 3 days.
- Storage temperature should be similar or inferior to the digestion temperature.
- When storage is prolonged for over 24 hours, it is usual to allow the temperature to drop progressively and then re-heat just before initiating consumption. There should only be two cycles of cooling and heating as the repetition of this process can accelerate the degradation of the rubber and thus a loss of rubberised binder properties.

In cases where the cooling-reheating cycles are unavoidable, or in cases when storage is required for over 3 days (when weather conditions are unfavourable for pavement laying) the fundamental parameters of the binder should be tested: penetration, softening point and elastic recovery. Viscosity will be tested in the case of high viscosity binders. In the case of slight deviations regarding the product specifications, the following is recommended:

- Use the rubberised binder as though it were a conventional bitumen and always in asphalt mixtures that are going to be used for areas that require lower technical demands.
- As an alternative, it is possible to include or add the product to a tank where the rubber concentration and/or polymers is higher than the original mixture and, in this way recover the properties that have been lost.

If all these guidelines are followed the quality of the binder and an absence of incidents can be ensured in the asphalt mixture plant.

"It is necessary to find a balance between the convenience of the product consumption immediately after its production and the need to operate with a specific storage capacity and regulation of the supply to the plant"

Formulations for Rubberised Binders

7

As mentioned before, one of the aims of this Guide is to give precise orientation regarding composition to allow the obtention of the most commonly used binders with rubber in Spain. This includes the requirements according to the Spanish specifications that appear in Article 212 of the General Specifications (PG-3) as well as the Circular Order 21/2007.

It is known that the characteristics of rubberised binders depend on the origin and the particle size distribution of the crumb rubber, the base bitumen and eventually the polymer. This does not only apply to penetration but also to the chemical characterisation. Furthermore, the type of mixer used may also affect certain properties of the final binder.

Due to the above, it is not possible to offer an optimal composition for all bitumens and rubbers and all the mixing equipment. Nevertheless, it is possible to provide some indicative formulation which does almost always comply with the specifications. However, it is necessary to carry out additional work in the laboratory in order to optimise this formulation regarding properties and also regarding production costs.

"The features of a rubberised binder depend on the ELT crumb rubber, the base bitumen and eventually the polymer. Furthermore, the type of mixer used may also affect certain properties of the final binder"

In this sense, a series of works have been carried out in the laboratory and are described here below and conceived so that the formulations proposed in the next sections are general and provide the broadest validity possible.

7.1 Characteristics and selection of material

Here below the characteristics of the materials used to carry out the laboratory work are detailed.

7.1.1 Characteristics and selection of the base bitumen

According to the bibliography and experience, high content of aromatic compounds facilitates the interaction between rubber and bitumen. For the experimental development, samples of pure bitumen 50/70 penetration, from four refineries were analysed. In order to carry out the selection, the following factors were considered: the origin of the processed crudes and the preparation process. In this sense, the bitumens used were varied, and hence the range of performance was wide enough to encompass 100% of the bitumens commonly used in Spain.

The most common method used for characterisation is fractioning, as described in the standard NLT-373/94. Through this method four different fractions are distinguished: maltenes, soluble in n-heptane, saturates non-absorbed in calcined alumina, naphthenic-aromatic or simply aromatics, that are absorbed in calcined alumina in the presence of n-heptane and that are desorbed with toluene, and polar aromatics also known as resins which are desorbed from the calcined alumina once the saturated fractions and the naphthene-aromatics become separate. This is carried out using toluene and trichloroethylene as eluents.

The results of the fractioning (NLT-373/94) from the four bitumens were as follows:

	Bitumen Refinery 1	Bitumen Refinery 2	Bitumen Refinery 3	Bitumen Refinery 4
(A) Asphaltenes, %	18.2	13.8	18.8	14.1
(B) Saturates, %	17.7	9.7	12.0	22.4
(C) Naphthenic-Aromatics, %	43.4	48.3	46.4	45.8
(D) Aromatic-Polar, %	20.2	28.0	22.3	17.7
(C+D) Aromatics, %	**63.6**	**76.3**	**68.7**	**63.5**

Table 4. Composition of the base bitumen

In view of the results, refineries 2 and 4 were selected as the bitumens offering both maximum and minimum values respectively regarding the total content of aromatics. With the bitumen from these two refineries, the rest of the laboratory work was carried out.

7.1.2 Characteristics and selection of crumb rubber

The main characteristics of crumb rubber, with influence on the properties of the bitumens, are, on the one hand, the composition (the polymer content and even the percentage of natural rubber) and the particle size distribution, and on the other hand, the production method.

For all practical purposes, the crumb rubber that is marketed in Spain is obtained using ambient grinding processes. In this sense, it was the only crumb rubber chosen to carry out this present work.

As for **particle size distribution**, 100% of the rubber used passes through the 0.8 mm sieve with over 35% retained in the 0.5 mm sieve. This is a common grading range in the market for asphalt modification.

Finally, regarding the **chemical composition**, the type of rubber, for practical purposes, depends on the origin of the tyre. The origin may be truck tyres or light weight vehicles with different percentages of natural and synthetic rubber. Taking this into account, two types of rubber were chosen with the grading mentioned before. The first type came from truck tyres and passenger car tyres equally divided (50/50) and the second type was obtained exclusively from the grinding of truck tyres (100/0). The composition of both products may be observed in ***tables 5, 6 and 7***.

As for the measurement of the non-rubber component, apart from the carbon black, an analysis of the ketone extract was carried out according to ISO 1407 and from which the following results were obtained:

	Plasticizer + additives %		
Origin of ELT crumb rubber	**# 1**	**# 2**	**Average**
50% truck + 50% passenger car	8.40%	7.92%	8.16%
100% truck	5.75%	6.01%	5.88%

Table 5. Analysis of the rubber ketone extract

The measurement of the main components of the crumb rubber was carried out with a thermogravimetric analysis using a thermobalance TGA/SDTA 851 Mettler and included the following conditions:

- Sample mass: 20 mg.
- Nitrogen flow: 80 mL/min.
- Oxigen flow: 45 mL/min.
- Testing temperature: 30 - 1,000 °C.
- Heating ramp: 20 °C/min.

The results can be seen in ***Table 6***.

	TGA			
Origin of ELT crumb rubber	**Volatiles %**	**Rubber %**	**Carbon black %**	**Ash %**
50% truck + 50% passenger car	4.67	57.41	32.22	6.02
100% truck	4.83	57.05	31.69	6.83

Table 6. Thermogravimetric analysis

For a complete characterization of the crumb rubber, measurements of the natural rubber content were carried out using the ^{14}C method. The results for both types of crumb rubber were as follows:

Origen of ELT crumb rubber	**% natural rubber**
50% truck + 50% passenger car	38%
100% truck	47%

Table 7. Natural rubber content

With these two types of crumb rubber, all the experimental laboratory work was carried out. In this manner, the formulations that are offered in the following sections are valid for rubber 100/0 (100% truck tyres) as well as for rubber 50/50 (truck tyres and passenger car tyres). Nevertheless, any other rubber samples obtained from a mixture of tyres and within these ranges should have a similar performance to those mentioned in this study.

7.1.3 Characteristics and selection of polymers

During the first laboratory tests, the most efficient modification obtained was when using a thermoplastic copolymer butadiene/styrene 70/30, polymerized in solution and with a radial structure. The trade name used is Calprene 411. This is presented in a porous crumb like form, pellet and powder with silica. In the laboratory, the powdered version were used due to the fact that the mixer used was a high speed propeller type without shear. Therefore, powder form is recommended in order to facilitate the integration with the bitumen.

7.2 Manufacturing protocol

The binders were manufactured using the equipment shown in ***figures 9 and 10 in Annex I***. This equipment consists of a high-speed propeller mixer and a 1 litre container/bowl in a thermal bath filled with oil. The sample quantity used is 650 g. Following a initial manual mix with a spatula, the mixing head is put in place. The container is sealed thanks to a perforated lid for the hand axis and an adhesive aluminium tape making it air-tight. After a ten minute rest period that allows the sample to reach the working temperature, the mixing procedure commences. Depending on the type of bitumen the precise speed needs to be selected. The details regarding temperature, mixing speed and time periods may be found in this Guide.

Once the rubberised binder had been prepared, samples were taken for the following tests: penetration, softening point and elastic recovery, as well as viscosity in the case of high viscosity rubber modified binders.

For the design of each rubberised binder, four samples were prepared:

- Refinery bitumen 2 + crumb rubber 50/50 (+ polymer in the manufacture of PMB binders).
- Refinery bitumen 2 + crumb rubber 100/0 (+ polymer in the manufacture of PMB binders).
- Refinery bitumen 4 + crumb rubber 50/50 (+ polymer in the manufacture of PMB binders).
- Refinery bitumen 4 + crumb rubber 100/0 (+ polymer in the manufacture of PMB binders).

For each one of these four combinations, several contents of crumb rubber were tested and in the case of the PMBs, several contents of polymers were tested.

Finally, and taking into account the results obtained, the most conservative compositions were selected in order to guarantee compliance in the four cases under study. All this while respecting other types of crumb rubber with gradings and contents of natural rubber much more favourable, as well as more compatible bitumens that could lead to formulations with higher percentages of rubber.

Using this manufacturing protocol the ideal composition was determined to produce binders BC 35/50, BC 50/70, PMB 45/80-60 C, PMB 45/80-65 C, BMAVC-1, BMAVC-3, and a final binder that is not included in the standard BMAVC-1b, softer than BMAVC-1, and whose used is recommended for cold regions.

"The most conservative compositions were selected to ensure compliance in the four cases under study"

The following sections introduce the type formulations for each one of the binders.

7.3 Formulations recommended for rubberised binders

In this section of the Guide, a series of formulations are recommended for different types of rubberised binders. To keep it simple, these formulations are presented in the form of manageable sheets. However, it should be noted that these are formulations that have produced good results with the materials and laboratory conditions mentioned previously. Therefore, they should be taken as approximate formulations that require verification in the lab and perhaps some adjusting in order to comply with required specifications.

7.3.1 Rubber enhanced binders

The rubber enhanced binders are compiled in the Circular Order 21/2007 by the Spanish Ministry of Public Works and Transport. These are binders with enhanced characteristics compared to bitumens with similar penetration, and with a higher softening point temperature. The standard nomenclature consists of the letters BC followed by the penetration range. There are two types: BC 35/50 and BC 50/70.

The recommendation is that it may be used in the manufacture of all bituminous mixtures, for any layer and traffic category that previously used paving grade bitumens.

The manufacturing process is simple due to the limited rubber content which results in a low viscosity value. However, there may be some settling, so the necessary measures should be taken during production and handling.

The recommended formulation for these binders is presented in technical ***sheets 1 and 2***.

SHEET 1

BC 35/50

Components	Production
Base Bitumen: B50/70	**Temperature (ºC):** 185
Rubber: 0/0.8 mm	**Type of stirrer:** propeller, 4,000 rpm
% Rubber (over bitumen): 10	**Digestion time:** 60 min
Manipulation and storage	
The product may decant. It is necessary to use tanks with a stirrer.	

SHEET 2

BC 50/70

Components	Production
Base Bitumen: B70/100	**Temperature (ºC):** 185
Rubber: 0/0.8 mm	**Type of stirrer:** propeller, 4,000 rpm
% Rubber (over bitumen): 10	**Digestion time:** 60 min
Manipulation and storage	
The product may decant. It is necessary to use tanks with a stirrer.	

7.3.2 Rubber modified binders

In the case of these binders, part of the modification is achieved with rubber since polymers are also frequently included. The technological characteristics are specified in article 212 of the Spanish Specifications PG-3. This article focuses on polymer modified binders and establishes that in the case that the majority of the modification is obtained using crumb rubber from tyres, at the end of the nomenclature the letter "C" must be added to indicate the use of rubber. In this sense, a binder PMB 45/80-60, with a paving grade between 45 and 80 tenths of a millimeter, and softening temperature of 60ºC or higher, has been achieved using mostly crumb rubber from tyres is then named PMB 45/80-60 C.

In this Guide, two rubber modified binders are presented: PMB 45/80-60 C and PMB 45/80-65 C. By adding the letter "C" it is made clear that these binders contain a larger proportion of tyre rubber.

These binders may be used for bituminous mixtures, layers and traffic categories in which bitumens that have been modified with polymers are recommended.

It should be noted that these binders may decant, and therefore the necessary measures should be taken during production and handling so that this does not occur.

The formulations of these binders are offered in *sheets 3 and 4.*

SHEET 3

PMB 45/80-60 C

Components	***Production***
Base Bitumen: B110/120 **Rubber:** 0/0.8 mm **% Rubber (over bitumen):** 4 - 5 **% Polymer C411 (over bitumen):** 2.5 - 3	**Temperature (ºC):** 185 **Type of stirrer:** propeller, 8,800 rpm **Digestion time:** 60 min

Formulation

Depending on the aromatic content of the base bitumen, laboratory tests will be required to determine the optimal dosage.

Production

A mixer equipped with shears is recommended for production.

Manipulation and storage

The product may decant. Tanks with stirring equipment will be required.

SHEET 4

PMB 45/80-65 C

Components	Production
Base Bitumen: B110/120 **Rubber:** 0/0.8 mm **% Rubber (over bitumen):** 4 - 5 **% Polymer C411 (over bitumen):** 3 - 4	**Temperature (ºC):** 185 **Type of stirrer:** propeller, 8,800 rpm **Digestion time:** 60 min

Formulation
Depending on the aromatic content of the base bitumen, laboratory tests will be required to determine the optimal dosage.

Production
A mixer equipped with shears is recommended for production.

Manipulation and storage
The product may decant. Tanks with stirring equipment will be required.

7.3.3 High viscosity rubber modified binders

High viscosity rubber modified binders are included in the Circular Order 21/2007 from the Spanish Ministry of Public Works and Transport and constitute binders that are highly modified. In this Guide, instructions are given regarding the manufacture of two of the most commonly used binders: BMAVC-1 and BMAVC-3. The first binder is obtained with high percentages of rubber and has a high viscosity level. The second binder, however, is manufactured with rubber and polymers, and eventually other additives. This binder is less viscous than the first binder but higher in values compared to more conventional bitumens.

The Guide also introduces a third type of high viscosity binders which has been named BMAVC-1b. The letter "b" indicates that it is a softer version of the BMAVC-1. The reason for including it in this Guide is because, after approval of the Circular Order 21/2007 it was found that the BMAVC-1 did not provide the flexibility required in areas with very cold winters. In this sense, a similar binder was applied successfully but made with a softer base bitumen. This one has been called BMAVC-1b.

High viscosity rubber modified binders are especially recommended when a high resistance to cracking is required, especially regarding heightening of semi-rigid paving and concrete slabs.

These are binders with a sedimentation tendency and therefore the necessary measures should be taken during production and handling.

The formulations of these binders appear in *sheets 5, 6 and 7.*

SHEET 5

BMAVC-1

Components	Production
Base Bitumen: B35/50	**Temperature (°C):** 195
Rubber: 0/0.8 mm	**Type of stirrer:** propeller, 4,000 rpm
% Rubber (over bitumen): 22	**Digestion time:** 60 min

Formulation

With certain base bitumens, with lower content of aromatic fractions, the rubber content will be around 20%.

Manipulation and storage

The product may decant. Tanks with stirring equipment will be required.

SHEET 6

BMAVC-1b

Components	Production
Base Bitumen: B50/70	**Temperature (°C):** 195
Rubber: 0/0.8 mm	**Type of stirrer:** propeller, 4,000 rpm
% Rubber (over bitumen): 22	**Digestion time:** 60 min

Formulation

With certain base bitumens, with lower aromatic fractions, the rubber content will be around 20%.

Manipulation and storage

The product may decant. Tanks with stirring equipment will be required.

Explanatory note

This binder is not included in the Circular Order 21/2007. It is a softer bitumen and has shown improved performance in colder areas compared to the BMAVC-1.

The basic properties of this kind of binders are:

- Penetration (EN 1426): 30/40 10-1mm
- Softening Point (EN 1427): ≥ 68°C
- Dynamic viscosity at 170°C (EN 13302): ≥ 2,000 cP
- Elastic recovery at 25°C (EN 13398): ≥ 10%

SHEET 7

BMAVC-3

Components	Production
Base Bitumen: B110/120 **Rubber:** 0/0.8 mm **% Rubber (over bitumen):** 4 - 5 **% Polymer C411 (over bitumen):** 3.5 – 4.5	**Temperature (°C):** 185 **Type of stirrer:** propeller, 8,800 rpm **Digestionn time:** 60 min

Formulation

Depending on the aromatic content of the base bitumen, laboratory tests will be required to determine the optimal dosage.

Manipulation and storage

The product may decant. Tanks with stirring equipment will be required.

The results of the analyses are summarized in the following table:

	Base Bitumen	Rubber content [(1)] (%)	Polymer content [(2)] (%)	T_{mixing} (°C)	Agitation level [(3)]	Digestion time (min)
BC 35/50	B50/70	10	-	185	Intermediate	60
BC 50/70	B70/100	10	-	185	Intermediate	60
PMB 45/80-60C	B110/120	4 - 5	2.5 - 3	185	High	60
PMB 45/80-65C	B110/120	4 - 5	3 – 4	185	High	60
BMAVC-1	B35/50	22	-	195	Intermediate	60
BMAVC-1b	B50/70	22	-	195	Intermediate	60
BMAVC-3	B110/120	4 - 5	3.5 – 4.5	185	High	60

(1) Over the weight of bitumen
(2) Over the weight of bitumen
(3) Intermediate stands for a propeller. To achieve a high agitation level a mixer with shear effect is required

Table 8. Formulations recommended for rubberised binders

Equipment for the Manufacturing of Rubberised Binders in the Laboratory

ANNEX 1

There are different apparatus and equipment for the production of rubberised binders in the laboratory; from simple mixers right up to the most sophisticated pilot plants which include computer control of the main production parameters.

In this annex, some general guidelines are given as well as a description of several cases and the frequently used equipment for the manufacture of rubberised binders.

Types of mixers

Two types of mixers may be used to produce rubberised binders:

- those that apply a more or less vigorous process of agitation and,
- those that have a shearing mechanism that, apart from mixing the components, also cuts the rubber particles making them much smaller.

Taking into account that there are numerous companies and types of equipment, the difference between one type and the others is not always discernible. On the one hand, there are mixers with a propeller that continuously stirs the bitumen and rubber in order to promote the interaction and prevent settling.

These devices, depending on the design and type of tank, can spin at a velocity of up to 1,000 rpm.

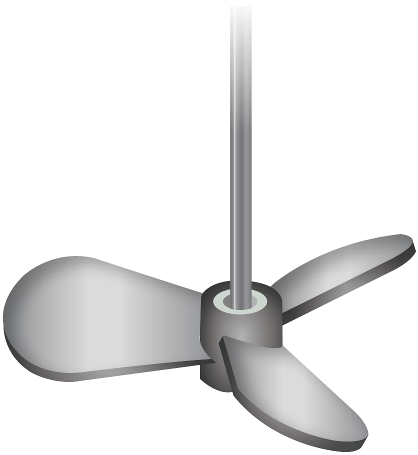


Figure 7. Head of a propeller mixer

On the other hand, there are mixers with shears that are usually made up of a fixed piece (stator) and a moving piece (rotor) with a small clearance between both pieces. This configuration produces a suction effect and therefore forces the rubber-bitumen mixture to pass between the two pieces and ensures an intense cutting effect which helps to reduce the rubber particle size. This increases the specific surface and promotes interaction between the rubber and the bitumen.

Figure 8 shows the mixer head with shear effect and the different stages that take place in the apparatus during the mixing process.

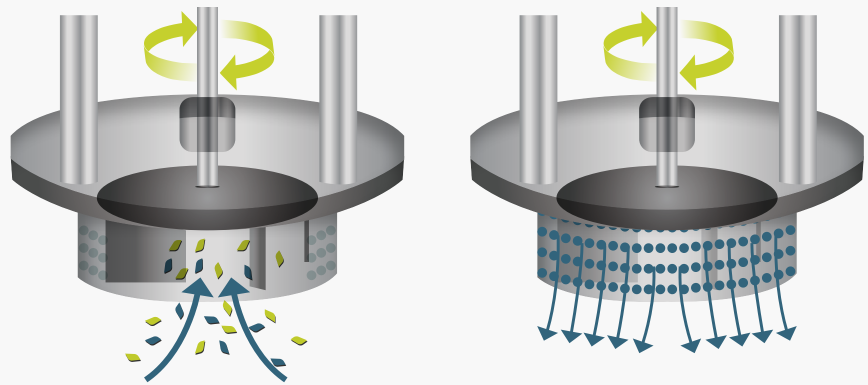


Figure 8. Mixer head with shear effect

There are different models of mixers and depending on the type of head and the speed selected these may function as mere mixers or include a shearing effect. When a propeller mixer is used at high speed, between 1,000 y 10,000 rpm, a dynamic effect is created and the particles are cut up.

Three different types of equipment are presented in the next section. These may be used in the laboratory to manufacture rubberised binders.

1. High-speed propeller mixer

This is one of the simplest devices that only achieves continuous agitation or stirring of the bitumen-rubber mixture. However, by increasing the speed some of these devices are able to go beyond just stirring. This is the case of apparatus that use a propeller and can reach speeds of up to 10,000 rpm. These devices can be seen in *Figure 9*.

Figure 9. Laboratory mixer with speed range between 500 and 10,000 rpm

In order to produce rubberised binders based on this scheme, it is necessary to carry out consecutive mixes by introducing the accessory into a metallic container which has been placed in a thermal bath of oil. *Figure 10* shows an example of this set up.

Figure 10. Setup of the basic laboratory equipment used to manufacture rubberised binders

It is very important to ensure that air contact with the bitumen is minimized in order to avoid oxidation of the product. This may be achieved by using a perforated lid so that the mixer head may be placed inside the container and reduce air contact during the 60-minutes mixing process.

As for the manufacturing parameters, it is necessary to select the temperature of the oil bath thermostat and the mixing speed using the dial that is included in the device.

The operation will be carried out in two phases: the first phase at high speed (3,000-10,000 rpm) in order to achieve a vigorous first mix. This will be followed by a second digestion phase while the apparatus works at a low speed (500-1,000 rpm) in order to avoid settling and to promote interaction between bitumen and rubber. The first phase lasts approximately 15 minutes while the second phase lasts for 45 minutes.

With the same laboratory equipment and trying to reproduce and get as close as possible to the industrial equipment where the mixer is also used as a digestion tank, it is viable to work at high speed during the 60 minutes. In any regard, the idea is to simulate as near as possible the conditions at industrial scale.

Generally speaking, the mixture is prepared in a 1 litre metallic container. However, this must not be completely filled with bitumen as it may overflow once the machine head is placed inside. Furthermore, it is necessary to allow for some space between the bitumen and the lid in order to facilitate handling. Therefore, experience shows that the sample to be kneaded should not be more than 650 g of rubberised binder. In the same way, the quantity should not be less than 650 g otherwise the machine head would not be covered.

In any case, a visual check should be carried out to ensure that depending on the capacity of the container and the mixer used there are no dead bands or areas that receive less agitation compared to the rest of the sample.

2. Laboratory mixing system with shear effect

These are mixers that include shears in the mixing head. The operation is similar to the previous examples except for the fact that the particles are subject to a much more effective cutting process. *Figure 11* shows an example of this apparatus and details of the head.

Figure 11. Laboratory mixer with shear

As in the previous case, a sample container is required and thermostatic oil bath to control the temperature during manufacture. The rotational speed may also be selected and can reach speeds of up to 8,000 rpm.

This type of equipment can manufacture the product in a single phase at high speed (4,000-8,000 rpm) or in two separate phases; the first phase at high speed and a second phase at a lower speed (500-1,000 rpm). The decision to choose either one or the other will depend on the configuration of the industrial equipment. In any case, the sample must be continuously agitated at a high temperature for 60 minutes until digestion is complete.

It is also necessary to avoid as far as possible the contact with air in order to reduce the oxidation of the bitumen during production.

The kneading in this type of mixer is also about 650 g, produced in a metal container with a capacity of 1 litre.

3. Pilot Plant for the manufacturing of rubberised binders

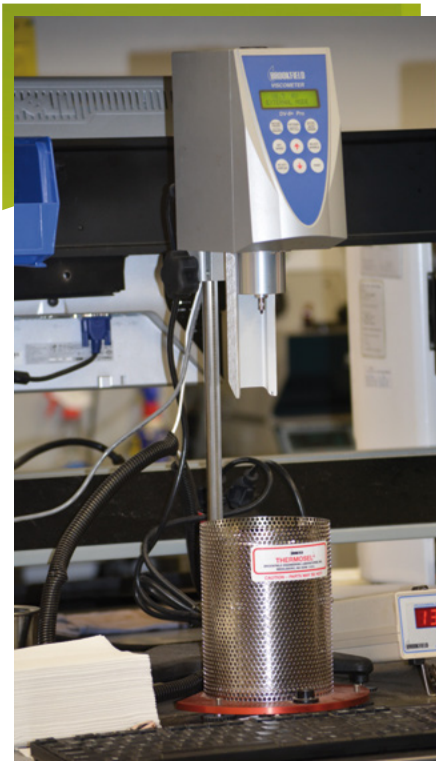
Likewise, there are laboratory pilot plants for the manufacture of rubberised binders that use the shearing system. Given the difficulties of incorporating rubber into the bitumen, it is commonplace that these mixers work in line with re-circulation and therefore it is necessary to have an additional tank. The next figure shows one of these models that combines an in-line system with shears through which the content from the upper tank flows continuously due to the propeller mixer.

Figure 12. Pilot plant for the manufacturing of rubberised binders

These devices offer state of the art technology with direct control over the most crucial manufacturing parameters. They usually require an external heater in order to maintain adequate temperature using heating jackets that cover the principal elements.

Another significant advantage is that within the circuits, the sample has no air contact. Nevertheless, it is necessary to take the same precautions explained previously regarding the upper tank.

One of the disadvantages is that regular cleaning is required and this is more difficult compared to case 1 and 2, where the container is discarded after each batch and where there are no circuits and therefore no obstructions caused by the bitumen. Furthermore, the price of this third option is considerably higher than the first two devices.



Recommendations regarding equipment to be used in the laboratory

It is not easy to make a specific recommendation regarding the equipment to be used in the laboratory, however, certain guidelines can be given and taken into account before making a decision:

1. In order to best reproduce the industrial process, the laboratory equipment should have an operating system (kneading or in-line, with or without shears) in accordance with the industrial equipment to be used.

2. For the majority of rubberised binders, a propeller mixer is usually enough as long as it is able to operate at medium to high speeds; above 3,000 rpm.

3. When polymers are used together with rubber, it is convenient to use equipment with shears or at least blenders that are capable of reaching high speeds above 8,000 rpm.

4. The decision should also be based on the type of use; for moderate use, the first alternatives 1 and 2 should be sufficient. The price of this equipment is 4 times less than a pilot plant, and routine cleaning is much easier.

Equipment for the Manufacturing of Rubberised Binders at Industrial Scale

ANNEX 2

Industrial equipment for manufacturing rubberised binders must have, from a functional point of view, two clearly defined units: on the one hand the mixer and on the other the digestion tank. In the first unit, the rubber bitumen mixture is obtained as well as a reduction in particle size, if the mixer is equipped with shears. In the digestion tank, the interaction between the rubber particles and the bitumen takes place at high temperatures. This is promoted by the stirring system that comes equipped with the tank.

From this basic standpoint, there are several different configurations: mixers with shears or without shears, independent mixer and digestion tank; or both systems together and even auxiliary tanks for storage and therefore the capacity to regulate the supply of rubberised binder to the worksite. These tanks are able to store rubberised binder until required by the production plant for asphalt mixtures.

In the next section, the most frequently used types mixers available on the market are accounted for, and through case studies, several configurations of manufacturing units are explained. Furthermore, an operation scheme is offered for the implantation of the unit in the asphalt mixture plant.

Paddle mixer

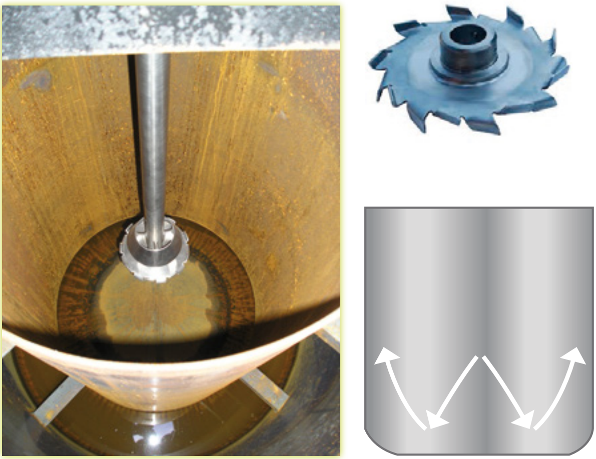
For binders with a low rubber content, below 10 %, paddle mixers or endless screw mixers should be sufficient. These devices provide the mixture with constant stirring (<1,000 rpm) so that the rubber is in suspension during the necessary period to complete the digestion process. The tank serves simultaneously as a mixer and digester. With such a simple layout, the rubber particle size should be very fine with a maximum of 0.5 mm. Furthermore, in cases, it will be necessary to prolong production time in order to ensure digestion, well over the usual 60 minutes period recommended when the system provides a more vigorous mix.

This system completely lacks the shearing system and is only found in devices for smaller productions. Use is not recommended due to the reduced amount of mixing energy and low production. *Figure 13* shows an example of the shaft paddle mixer.

Figure 13. Paddle mixer

Cowles disc mixer

This type of mixer transmits a great deal of energy (>3,000 rpm) due to its design and rotation speed and even creates a shearing effect. *Figure 14* offers an example of the Cowles disc and the position inside the bitumen mixer.



> ***"The use of the Cowles disc mixer is frequent due to the high hourly output and simple maintenance"***

Figure 14. Cowles disc mixer

This type of mixer produces a vortex which forces the rubber bitumen mixture to pass through the Cowles disc over and over again. In this way, the particles are cut smaller and smaller progressively, and the mixture becomes homogeneous.

This system can be installed in mixers but also in tanks which are used for mixing as well as for digestion. The production rate is high and maintenance is simple. This system is widely used for the manufacturing of rubberised binders.

Mixers with a rotor-stator shearing system

Without a doubt, this is the most evolved type of mixing system that submits the rubber and bitumen mixture to a high stress level due to the high speed of the shearing mechanism. Between the fixed section and the mobile section there is a slight clearance through which the rubber-bitumen mixture passes.

The blades cut the rubber particles until they are small enough to pass through the clearance. This type of mixer is known as Polymill, and far from being installed in a tank, these often work in line, recirculating the rubberised binder from the tank that also has a stirring paddle system. *Figure 15* shows an image of this type of mixer.

Mixers constitute the critical component of the rubberised binder manufacturing process. And these mixers may use a variety of configurations.

The most common configurations

Through a case study, the most frequently used configurations are presented.

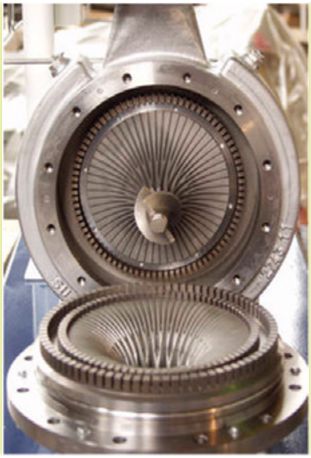


Figure 15. Mixer rotor-stator with shears

Configuration 1. Small volume mixer and a digestion tank

In this case, the two functions required to carry out production are separate:

First of all, a vigorous mix is carried out in a mixer equipped with a Cowles disc. From here, the product moves on to a larger digestion tank where the rubberised binder continues to be stirred with a shaft paddle mixer or with an endless helicoidal agitator until the required 60 minutes digestion period is complete.

The equipment is completed with hoppers for the storage and dosage of the crumb rubber and for any polymers or additives, pumps and even a heater in the case of mobile units so that they may function independently. In this way, it is not necessary to connect the thermal oil circuit used in the asphalt mixture plant.

Figure 16 shows this type of unit. In this example, a mobile unit which can move to the manufacturing plant and supply the rubberised binder and withdraw once the work is finished. In other cases, the unit uses the same configuration but it is stationary and remains in the asphalt mixture plant.

Figure 16. Manufacturing unit with mixer and digestion tank

As for the integration in the asphalt mixture plant, these types of units obtain pure bitumen from the storage tanks. It is then mixed with the crumb rubber and then dispensed on demand to the mixing plant through a pump which is operated from the control panel of the plant.

Figure 17 provides a schematic illustration of the implementation, where it is possible to see that this type of installation does not substantially affect any of the principal elements of the asphalt mixture plant.

The crumb rubber is usually provided in big-bags with an approximate capacity of 1 tonne (2 m^3) which is unloaded into the hopper of the mixing unit from which the crumb rubber is then measured out (see *figure 18*). In permanent or fixed installations bulk supply is also possible and may be stored in vertical storage silos equipped with a dispenser unit.

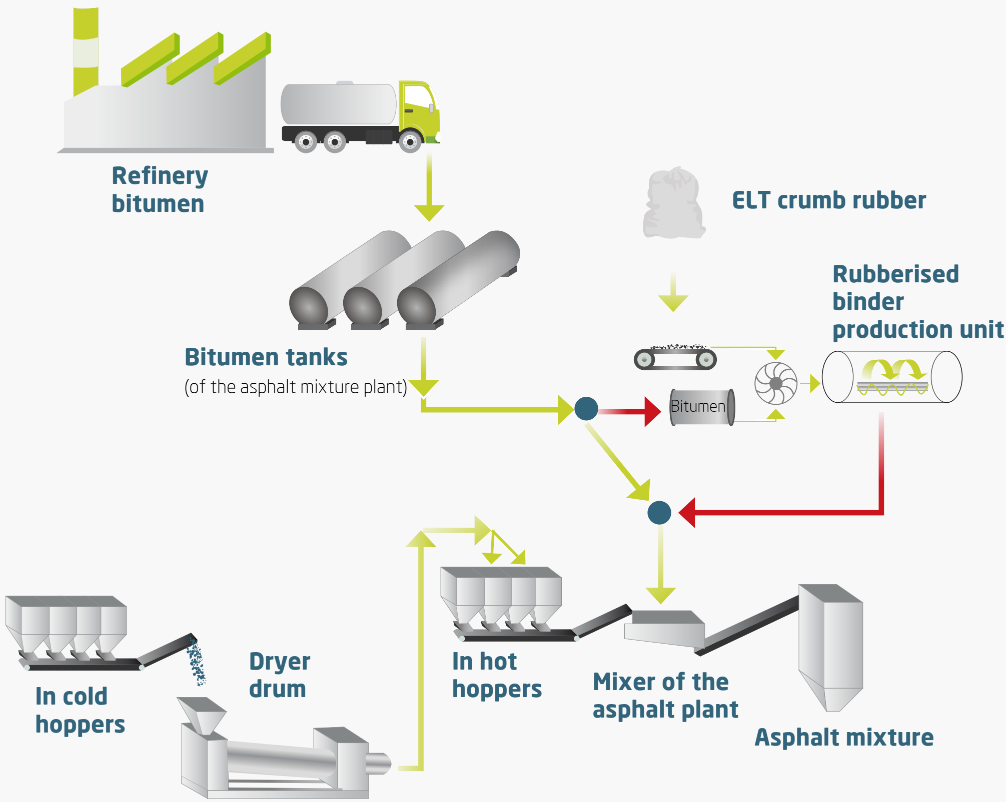


Figure 17. Implantation of the unit in an asphalt mixture plant

Figure 18. Detail of the loading of crumb rubber into the hoppers

As for the hourly output (tonnes/hour), these units coincide with the capacity of the digestion tank. If the digestion tank has a capacity of 15 tonnes, the hourly output will also be of 15 tonnes of rubberised binder.

Configuration 2. Twin tanks for mixing and digestion

In this particular case, the mixing and the digestion process takes place in the tank. Nevertheless, for efficiency and in order to achieve a higher hourly production the scheme is duplicated with two twin tanks so that while one supplies the 60-minutes digested binder, the second tank can produce the new batch of binder. In this way, one tank produces the rubberised binder while the other supplies the product to the asphalt mixture plant. A diagram may be seen in ***figure 19*** as well as in ***figure 6***.

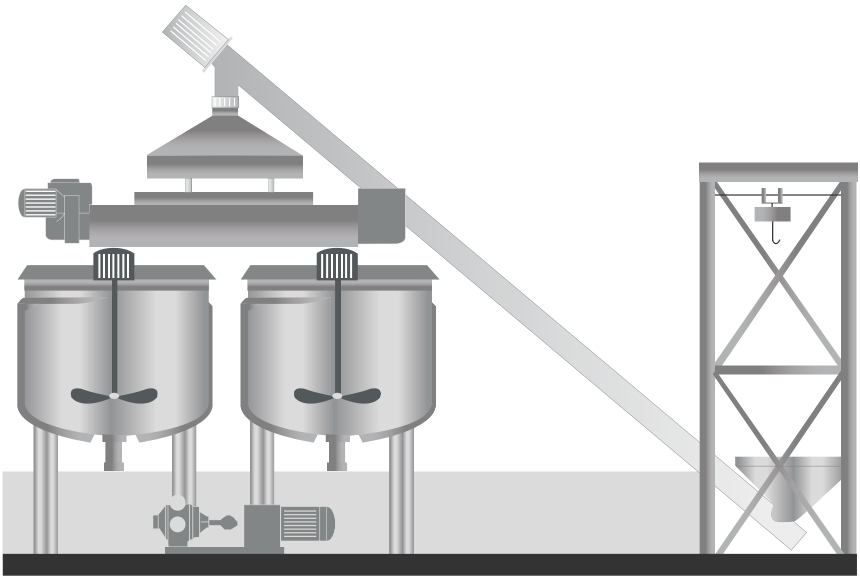


Figure 19. Outline of a unit with twin tanks

The tanks are usually equipped with a Cowles disc and additionally, depending on the design may be equipped with a shaft paddles mixer.

In the same way, as in configuration 1, this type of unit may be stationary or mobile and the implementation in the asphalt mixture plant is also similar. The hourly capacity (tonnes/hour) is usually around 150% of the tank´s capacity and will ultimately depend on the filling speed and other aspects concerning the design of the production cycle which is established by the manufacturer of the unit. In a similar manner, the capacity will depend on supply conditions at the worksite.

Configuration 3. Unique tank units with polymill in the recirculation line

This outline, which is typical of traditional plants where bitumen is modified with polymers, consists of a tank in which bitumen and rubber are incorporated and constantly remains in movement by means of a shaft paddles mixer. The content of the tank is kept in recirculation by a circuit in which a high speed mixer has been included: rotor-stator type with shear effect. There will be continuous recirculation of the product until the desired homogeneity is achieved and then be used. The previous cycle will then commence once again.

The unit is shown in ***figure 5*** of this Guide, where it is possible to observe the way in which the mixer is inserted into the line of recirculation.

This configuration is typical of permanent plants. The hourly production is around the capacity of the tank since a batch mix is produced every 60 minutes in order to ensure digestion. Nevertheless, the hourly production could be increased in the case that the product requires transportation to the asphalt mixture plant where it will be used as the transport period could be used to complete the digestion period.

Minimum requirements for a rubberised binder manufacturing unit

In order to ensure the quality and homogeneity of the rubberised binders manufactured by any of the production units mentioned above, special attention must be paid to dosing, mixing, in addition to carry out routine quality controls on the product. In this manner, the units should at least be equipped with the following elements:

- A weight dosage meter or device for the crumb rubber and the polymer.
- A weight/volume dosage device for the bitumen.
- Thermometers placed:
  - » at the entry point of the base bitumen
  - » inside the mixer
  - » inside the digestion tank, if it is not included in the mixer
  - » at the rubberised binder delivery line to the asphalt mixture plant
- Viscosimeters placed:
  - » in the digestion tank
  - » at the rubberised binder delivery line to the asphalt mixture plant (there are in-line viscosimeters)
- Taps or faucets for sample taking at the outlet of the digestion tank

Auxiliary storage tanks

Although any rubberised binder production unit should ensure that the product reaches the required degree of digestion, which takes place after 60 minutes and includes mixing as well as digestion, it might be convenient for logistic reasons to have auxiliary tanks for storage of the manufactured rubberised binder. These tanks should not be confused with the digestion tanks of the rubberised binder production unit.

When auxiliary tanks are available, the production unit delivers the rubberised binder to the storage tanks instead of directly to the weighing device at the asphalt mixture plant where it is offloaded into the mixer.

The advantages of these auxiliary tanks are the increased control capacity regarding the supply of binder to the asphalt mixture plant and the possibility to solve any problem that may arise in the rubberised binder production unit owing to the binder that is stored in the auxiliary tanks.

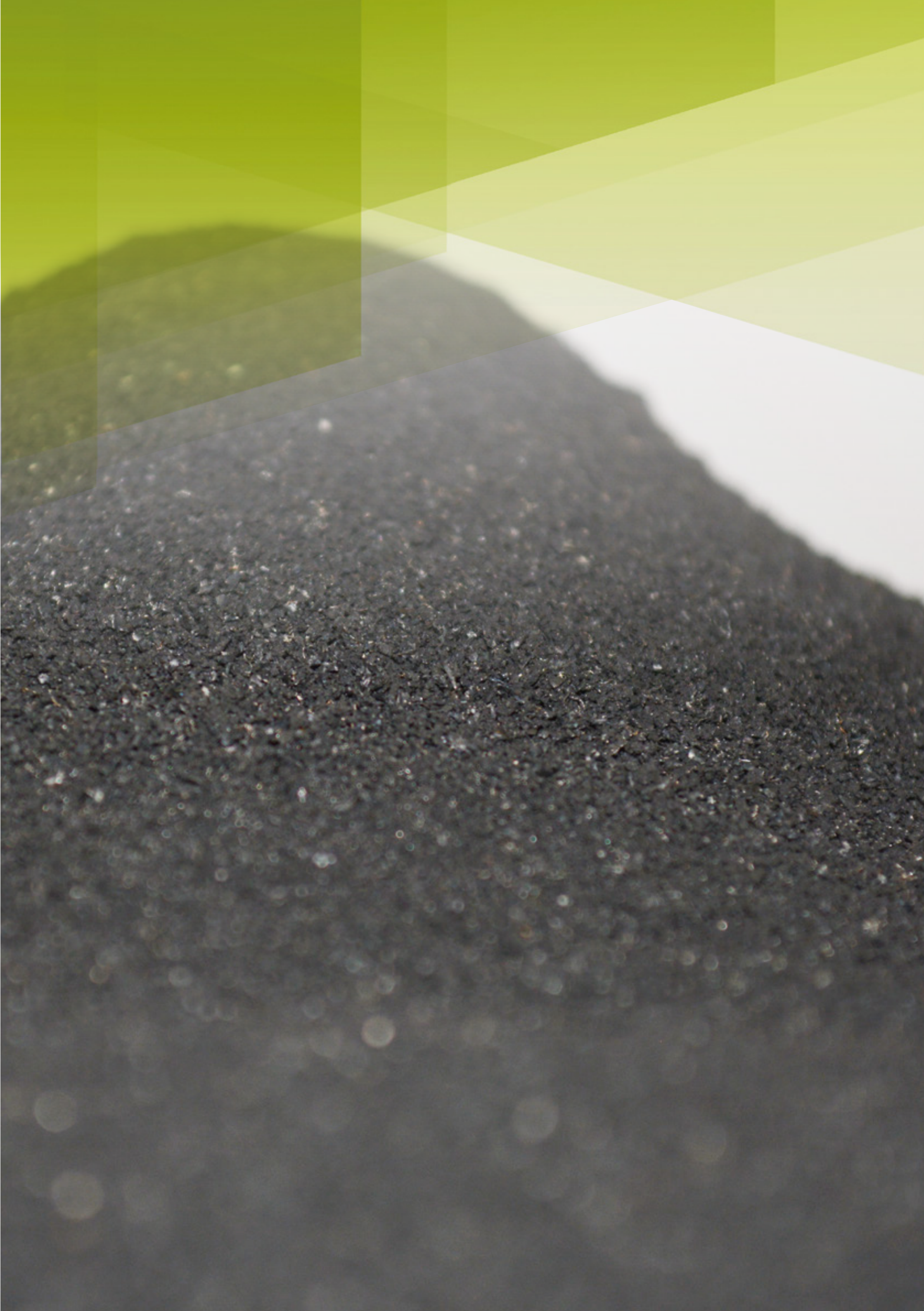
To ensure compliance with the storage requirements, these auxiliary tanks must be equipped with a heating system which enables the temperature control of the product during the hours or days necessary as well as a stirring system to avoid settling.

Figure 20. Auxiliary storage tanks

Choice of rubberised binder manufacturing unit

In order to choose the correct unit and taking into account the requirements of each asphalt mixture plant, the user should consider the following basic criteria:

- The paddle type mixer provides low energy to the mixture and is generally used for stirring purposes; this is, a way to maintain the product in movement while completing the digestion process. The units equipped with only this mixing device are not suitable for modified binders with polymers and crumb rubber.
- The units provided with a mixer and a digestor or with two twin mixing-digestion tanks are the most commonly used for the manufacturing of rubberised binders. These are both recommended and enable the production of rubberised binders as well as those bitumens modified with polymers. They are valid in most cases; however, certain tests are necessary initially to determine whether the polymer to be used should be in powder form or if it is possible to use pellets or granules.
- The units provided with a shear device are the top-level regarding mixing units. These units can achieve excellent homogeneity as they re-circle the product as many times as required until reducing the particle size. Furthermore, in the case of using polymers, these may be used in pellets or granules without difficulty.
- In all of the cases mentioned above, the following should be taken into account:
    - » The hourly production of the unit in order not to limit the asphalt mixture plant.
    - » If the production unit is autonomous or requires thermal oil supplies or electricity from the asphalt mixture plant.
    - » The area that the unit occupies on the asphalt mixture plant and how this may affect the operability of plant: loading and unloading of trucks that transport the asphalt mixture, access to tanks when delivering or supplying base bitumen, etc.



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