Binder performance and testing

GLOBE: an innovative technical solution to ensure waste free cold logistics of bituminous binders

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Abstract

GLOBE is a French acronym for "Granulés pour la LOgistique des Bitumes d'Enrobage" literally meaning « Bituminous pelletsfor the logistic of coating binders ». This project supported by ADEME (French Environment & Energy Management Agency) focuses on the development of an innovative technical solution to ensure a logistic of bituminous binders from refineries to asphalt mix plants which is cold, waste free and thus cleaner and safer. The main challenge is to modify a material such as bitumen, usually handled hot in liquid form, in order to be able to produce a granular form of it, which stays stable over time. In order to do that, it is required to overcome in-depth its typical binder characteristics, especially its creep behavior and its exceptional adhesiveness properties. This implies to modify the rheology of the binders and to take into consideration the granulation technology. The final product should avoid agglomeration phenomena, while taking into account the mechanical and thermal stresses associated with the handling, storage and transport of the pellets. In addition, the characteristics of bitumen should be recovered after mixing and laying, namely, the adhesion to the aggregates to insure the cohesion of the granular skeleton and the mechanical characteristics to guarantee the transfer of the mechanical stresses within the asphalt material during the lifetime of the infrastructure. This project will also be subject to environmental monitoring, as it is important to check that the apparent gain is real throughout the entire life cycle. Key words: bitumen, pellets, rheological properties, granulation technology, ACV

This paper was first published in 2020 in version 1.0 of the proceedings of the 7th E&E Congress. This second edition (version 1.1.) of the proceedings is published in June 2021. The content of this paper has not changed, but the author(s) may have provided additional information in the form of an annex.

1. SCOPE OF THE STUDY

1.1. GLOBE solid bitumen approach: benefits & challenges toward current ecosystem

Most of bitumen material is used in building and road pavement application. Bitumen is generally in the form of a black material which is highly viscous or even solid-like at ambient temperature, and which liquefies when heated. But from a rheological point of view, common paving grade bitumen is still considered as a viscoelastic liquid above ambient temperature (temperature crossover point).

Generally, bitumen is stored and transported under hot conditions, in bulk, in tanker trucks or by boats at high temperatures of about 120°C to 160°C.

However, the storage and transportation of bitumen under hot conditions has some drawbacks. Firstly, the transportation of bitumen under hot conditions in liquid form is considered to be dangerous and is highly restricted from a regulatory point of view. This mode of transportation does not present any major difficulties when the transportation equipment and infrastructure are in good conditions. If this is not the case, it can become problematic: for example if the tanker truck is not sufficiently lagged, the viscosity of the bitumen will increase as the temperature drops during a long trip and offloading can become difficult. Bitumen delivery distances are therefore limited. Secondly, maintaining bitumen at high temperatures in tanks or in tanker trucks consumes energy. Lastly, maintaining bitumen at high temperatures for long periods can affect the properties of the bitumen and thus change the final performance levels of the asphalt mix.

In order to overcome the problems of transporting and storing bitumen under hot conditions, packaged solutions for transportation and storage for bitumen in solid form under "ambient" and cold conditions have been developed. This mode of transportation of bitumen in such conditions represents only a minimal fraction of the amounts transported throughout the world, but is essential for some geographic regions which are difficult and expensive to access using conventional transportation means.

As an example of "cold logistic", mention may be made of transporting bitumen at ambient temperature in metal barrels. This is the most common way of transporting bitumen at ambient temperature.

However, the use of metal barrels is increasingly questionable from an environmental point of view. Firstly because the cold bitumen stored in the barrels must be reheated before it is used. Secondly because the barrels will constitute a waste after use. Thirdly, handling solid bitumen in barrels results in losses as the bitumen is very viscous and part of the product always remains on the walls of the barrel when the bitumen is transferred into the tanks of the mixing production units. Lastly, the handling and transportation of bituminous products in these barrels can be difficult and dangerous if the specialized equipment for handling the barrels is not available in the asphalt mix plants where the bitumen is used.

Another example of "solid bitumen" may be made in the form of pellets transported in bags. These pellets have the advantage of being easy to handle. U.S. Pat. No. 3 026 568 describes bitumen pellets covered with a powdery material, such as calcium carbonate powder **[1]**. Nevertheless, such bitumen pellets tend to adhere to each other and to agglomerate during their storage and/or their transport, especially at relatively high ambient temperatures and over long periods.

Patent application WO 2009/153324 describes bitumen pellets coated with a polymeric anticaking compound, in particular polyethylene **[2].** The drawback of this coating is that it modifies the properties of the bitumen during its use phase on the road.

Patent application WO 2016/016318 describes bitumen pellets comprising a chemical additive. These bitumen pellets can be handled at ambient temperature without the bitumen undergoing creep, and without the pellets sticking together **[3].**

There is a need to provide a solid form of bitumen for easy handling and road pavement application, which can be transported and/or stored under wider conditions of temperatures and stress loadings, making it possible to overcome the drawbacks described above.

In particular, the aim of the present project is to develop a solid form of bitumen, which can be transported, handled and stored even at high ambient temperatures, especially at a temperature above 50° C commonly reached in containers in hot countries in order to set up a sustainable ecosystem based on handling solid bitumen under wider conditions as an opportunity for improving road pavement industry.

1.2. The technical challenges

In order to overcome the technical challenge of shaping a material such as bitumen, into a granular, solid form, stable over times, it is necessary to carry out in-depth modifications of its characteristics, in particular its flow capacity and its exceptional adhesiveness properties. However, the granular shape is desired only during the time of storage and transport of bitumen as the end use remains the same, namely: the cohesion of the granular skeleton and the transmission of mechanical stresses of the asphalt on the road.

To sum up the scope of the work, three scientific and technological barriers are identified with different levels of difficulties requiring the association of knowledge and skills in areas of formulation, industrial process and material sciences in order to reach:

-The possibility of shaping bitumen granules and of maintaining their dimensional stability

-The temporary removal of tackiness (natural stickiness) of the bitumen and the ability to not self-agglomerate.

-The recovery of the binder properties during the use in order to obtain performant pavement materials.

1.3. Pellet Design

In terms of industrial processes, obtaining the bitumen granules can be summed up in a granulation step ("pelletization", i.e. obtaining materials in granular form) followed by a step of encapsulation / filming (" coating ", i.e. obtaining a film) to ultimately result in a core/shell system.

There are a variety of technological processes for granulating and encapsulating organic materials. Several types of classifications are possible (depending on the size of the objects obtained, depending on the use or not of a solvent, depending on the nature of the constituents of the core and shell, etc...).

1.3.1. Modification of core to develop a "solid" bitumen

According to the literature, there are several ways to solidify a bitumen matrix **[4-8]**. Among them, the use of gelling additive at relatively low concentrations (< 5% mass.) have the advantage to get a solid bitumen over a range of temperatures, over a wide range of loading stresses (creep resistance) and over long time period and that without affecting the viscosity of the binder at processing temperature **[8].**

a) Methods of evaluation of the strengthening of bitumen modified with gelling additive

The modification of bitumen using gelling additive is relatively unusual and the conventional tests such as penetrability or R&B temperature are not suitable for capturing the specificity of its rheological / mechanical behavior. It is better to rely on specific tests to measure the solid behavior or its resistance to creep. So far, two tests developed during this project can be used to assess the physical properties of the bitumen formulations and that independently of the process parameters which significantly affect (positively or negatively) the real performances of the material (**Table 1**).

Table 1. Tests developed during this project to evaluate the characteristics of bitumen modified with gelling additive in terms of creep resistance

Test	Illustration	Description
Texture analyzer		Indentation test at imposed speed and at a fixed temperature (currently at 50°C). Measurement of the resistance force resulting from a dashpot displacement. Sample is conditioned in a standard penetration box.
Creep in compression		Creep test at imposed stress to estimate the critical stress at different temperatures. Typical block form of solid bitumen with thin layer of PE cover is used. The calculation of the height drop ratio gives the mechanical resistance of block of solid bitumen.

1.3.2. Formulation of a shell

Different families of coatings / releasing film agent have been identified: wax-type solutions, anti-caking agents (fillers, lime, and polymer microbeads), synthetic polymer matrices or bio-sourced used in paint coatings, in food industry, or in cosmetics but also in the pharmaceutical field. The main functions sought in the case of bitumen granules are the capacity to obtain a film eliminating the initial stickiness aspect of the bitumen and the capacity to insure the overall resistance of the bitumen granules during storage and transportation. In order to obtain such a film, it is necessary to formulate an appropriate coating solution. The quality of the solution will depend on the nature of the constituents, on the diffusion medium, on the application process and on the final targeted properties of the non-adhesive film.

The application of a uniform surface coating with a controlled thickness requires appropriate equipment. Film deposit can be done in different ways:

- By powdering a dusting agent,

- By coating a film-forming agent in the molten state,

- By coating a film-forming agent in aqueous phase.

1.3.3. Process of granulation/coating

Four kind of process based on different pelletization technologies are investigated to obtain GLOBE solid bitumen for this study. Process 1 and process 2 include a first step of granulation following by a second step of coating while a single step is required for process 3 and process 4 as it combines at the same time granulation and coating.

1.4. Solid bitumen storage stability evaluation

In order to mimic the conditions of storage in big-bag and to follow the thermomechanical abilities of pellets against global agglomeration (caking, clogging or loss of free-flowing behavior), pellets samples were conditioned into thin bags (150 g) before storing them on oven shelf as depicted in **Figure 1**. Then, a large metal plate was put on bags in order to guarantee stress loading distribution. Finally, big-bag equivalent loading stresses (6,5 kPa or 13 kPa) were applied by adjusting specific weights on the metal plate surface. Storage in oven at 50°C or 60°C were monitored during 2 months to define if the pellets of solid bitumen are prone to agglomeration or if they keep their original free-flowing abilities.

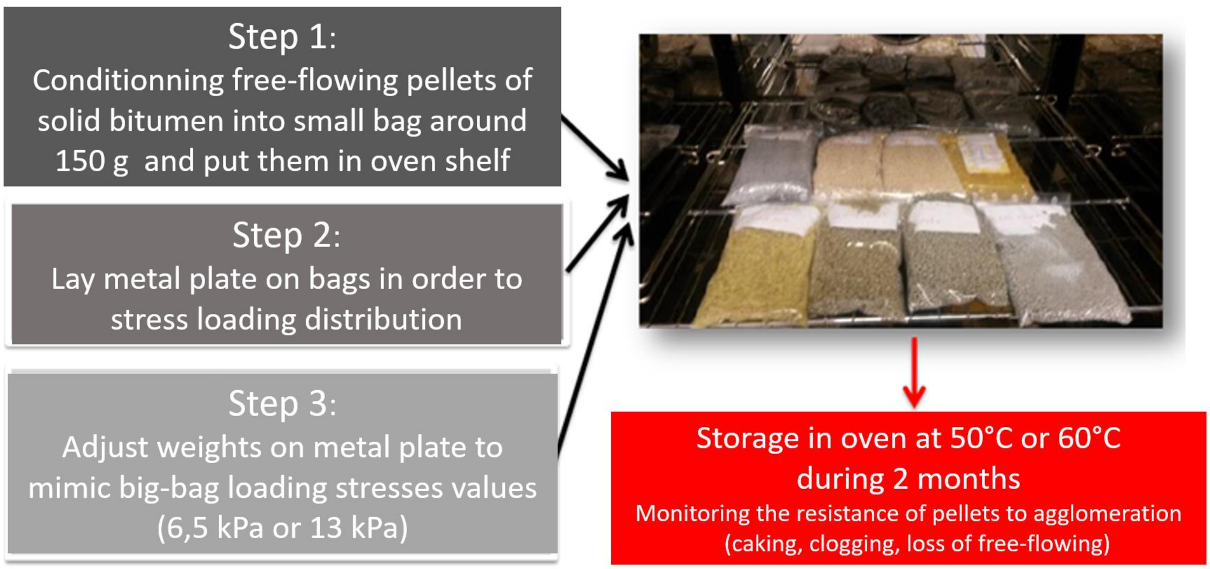


Figure 1 : Storage abilities evaluation of solid bitumen for global agglomeration and free-flowing

1.5. Asphalt mix evaluation

The end-use of solid bitumen have to be the same as the one of the current liquid form of hot bitumen in terms of asphalt mix manufacturing, laying aspects and overall pavement lifetime properties. In this paper, results were reported based on a preliminary comparison between asphalt mix made with some GLOBE solid bitumen and standard 35/50 paving grade bitumen.

Table 2 reported a typical surface course asphalt concrete mix (AC10) used for this investigation. For the binder content, strictly same equal mass ratio of 35/50 paving grade bitumen is replaced by two different formulations of GLOBE bitumen both based on process "3" but with two different contents of shell ratio (respectively 2% and 5%).

To evaluate the impact of pellet of solid bitumen on asphalt mix properties, standard evaluation methods as workability test using Giratory Compactor (PCG test), rutting test and Duriez test have been carried out to evaluate respectively the workability, rutting behavior and water debonding susceptibility **[9-11]**.

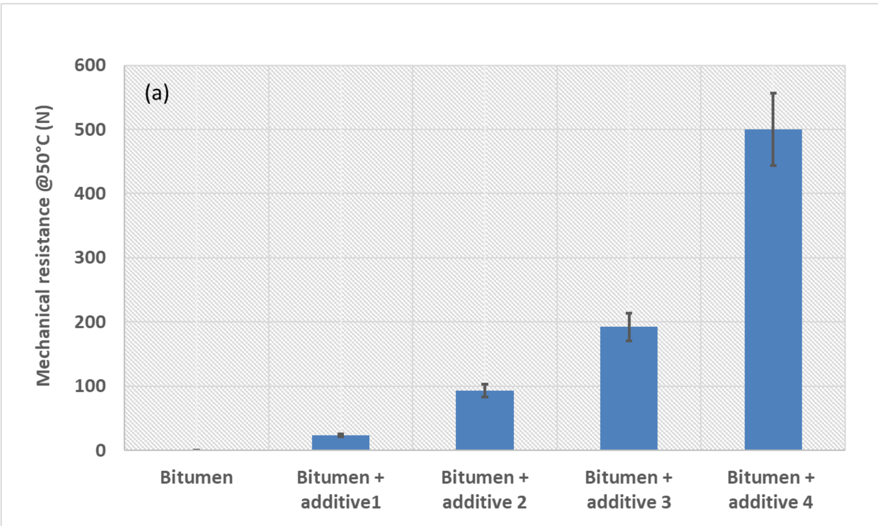
Table 2 : Asphalt mix design

Components	Percentage (%)		
6/10	51		
2/6	16		
0/2	24		
Filler	3.6		
35/50 paving grade bitumen	5.4	-	-
GLOBE bitumen (2% outer coating)	-	5.4	-
GLOBE bitumen (5% outer coating)	-	-	5.4

2. RESULTS AND DISCUSSIONS

Effect of gelling additive on solid bitumen mechanical resistance

The resistance force to deformation induced by a dashpot-like indentation is measured at 50 °C for bitumen modified with different types of gelling additive (including also standard 35/50 paving grade bitumen as control specimen) via the texture analyzer test shown in **Figure 2-a**. Although the absolute value is difficult to analyze (stress fields / non-uniform deformation, confined environment and wall effect), it is possible to compare and classify the relative performance of solid bitumen in function of the nature of gelling additive. For example, additives "3" and "4" exhibit the higher mechanical resistance while additive "2" seems to achieve maximum performance for a concentration around 1.2 - 1.5% (**Figure 2-b**). In the opposite, additive "1" does not display sufficient strength and consequently, the pellets of solid bitumen made with such additive will creep under severe stress loading and temperature.



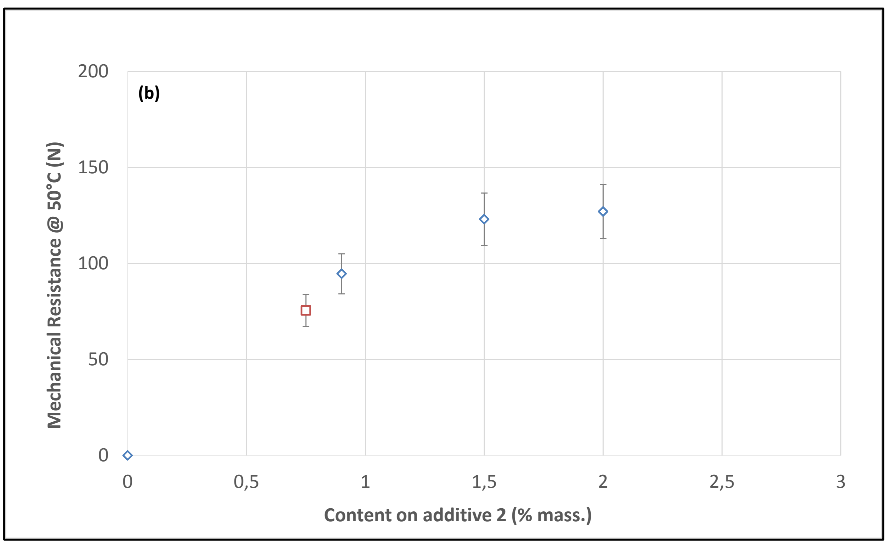


Figure 2 : Dependency of the mechanical strength of solid bitumen modified by different gelling additives (a) and versus ratio content of additive "2" (b).

In addition, **Figure 3** represents the behavior of the block of solid bitumen modified with gelling additive in function of temperature under a static stress loading of 6.5 kPa. A temperature above 70 ° C combined with a load of 6.5 kPa seems to be critical with the additive "1" as the mechanical strength loss is around 40%. It should be noted that the additive "3" has a better resilience than the additive "2" at 90°C.

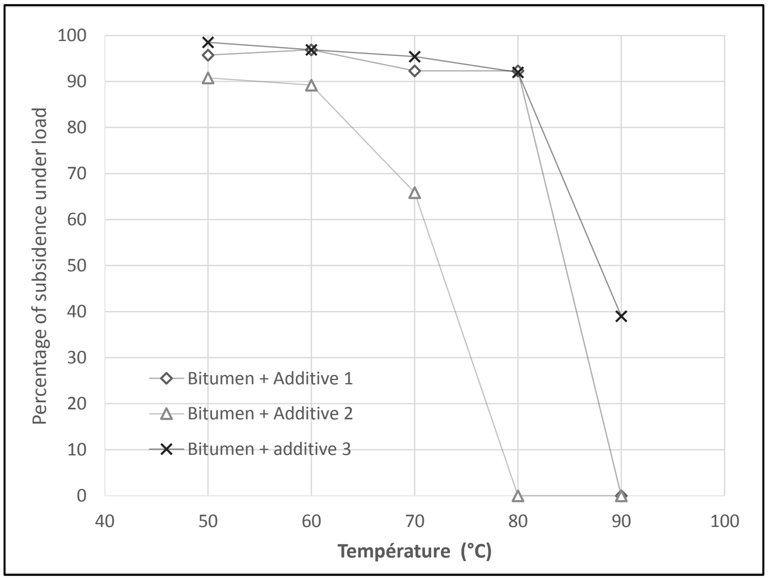


Figure 3 : Loss of the mechanical strength of solid bitumen in function of the temperature under a load of 6.5 kPa.

From these two specific tests, it is possible to screen the additives that will be the most appropriate to achieve the technical specifications of GLOBE bitumen in terms of creep resistance and storage abilities. It should be noted that this methodology for evaluating the performance of gelling additives is carried out without taking into account the positive /negative impact resulting from the process parameters, which will have to be evaluated and discussed hereafter.

Effect of process technology on storage abilities of solid bitumen

Figure 4. represents the maximum duration time of storage before agglomeration of pellet design (solid bitumen modified with additive "2") for the different process technologies investigated. At iso-modification of the core of pellet by additive "2", it is obvious that the process has an important effect on the stability and the performances of the pellets. For example, it is remarkable that the pellets obtained with process "1" can resist until 66 days under a load of 6.5 kPa at 50°C before pellet agglomeration. In opposite, pellets with the same solid bitumen modified with additive "2" but obtained with process "4" do not resist more than one day and lead to unacceptable agglomeration.

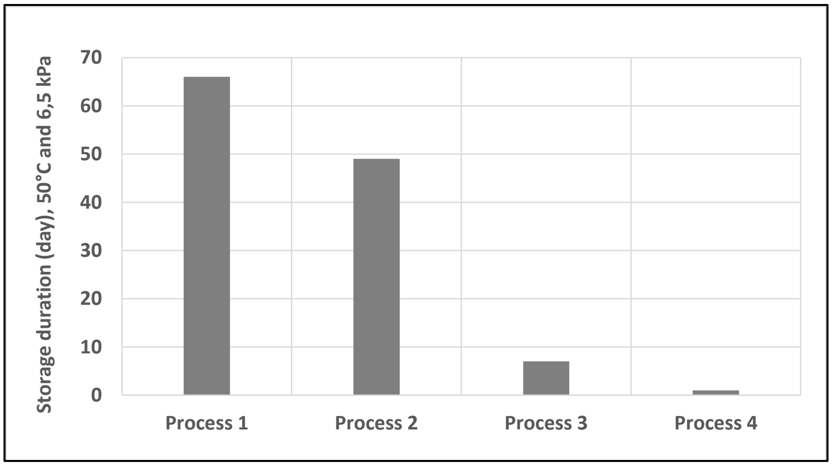
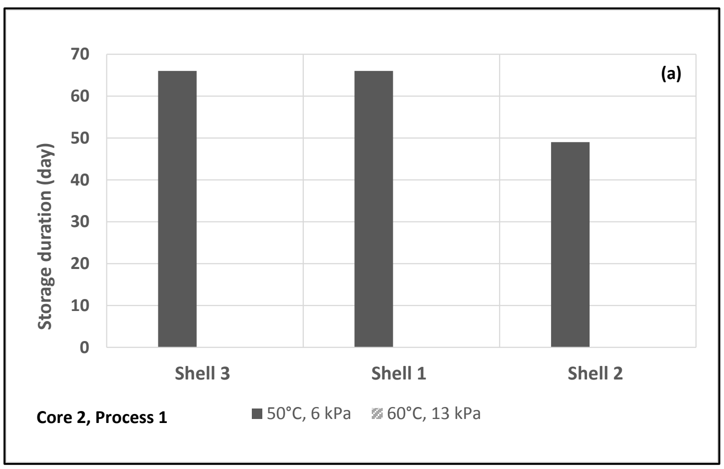


Figure 4 : Impact of process on the performances of pellets during storage with the same solid bitumen modified with additive "2".

Effect of the pellet design on the storage abilities

As process "1" is the most advanced route, the impact of the formulation of the shell has been investigated with two different natures of solid bitumen modified respectively with additive "2" and additive "4" for the formulation of the core of the pellet. **Figure 5** presents the impact of the nature of shell on the storage duration of the pellets under two conditions of storage (50°C, 6.5 kPa and 60°C, 13 kPa) before agglomeration.



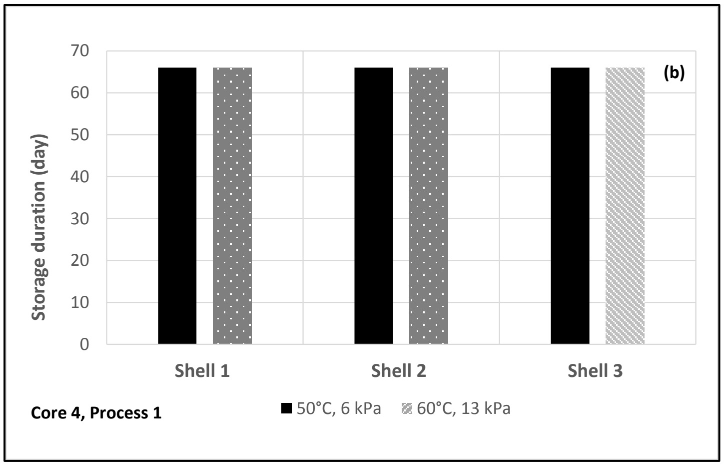


Figure 5 : Impact of the nature of shell on the storage quality of pellets with two types of core "2" and "4" and in function of stress loading (6,5 and 13 kPa) and temperature of storage (50 and 60°C). Full bar: any drop of free-flowing behavior, strips bar: some local agglomeration and slight drop of free-flowing behavior, drops bar: massive agglomeration and any free-flowing behavior

Figure 5-a shows that the pellets designed with shell "1" and "3" present the same performances. Such pellets resist under a storage of 66 days at 50°C and under 6,5 kPa without any agglomeration issues. However, at 60°C and under 13 kPa stress loading, one notes that pellet designed with the shell "3" leads to a higher storage duration time resistance before agglomeration than the ones obtained with shell "1". Unfortunetaly, after such severe storage conditions, pellet designed with shell "3" exhibits some shell crackings which lead to a drop of free-flowing. With bitumen modified with gelling additive "2", all pellets display a decrease of storage duration time as the storage condition severity increases.

Comparison between **Figure 5-a** and **Figure 5-b** shows that the nature of gelling additive in pellet core design has noticeable influence on storage abilities of pellets, additive "4" is most efficient than additive "2". Indeed, whatever the nature of shell, all pellets containing bitumen modified gelling additive "4" exhibit remarkable storage abilities at 50°C and 6.5 kPa. On the other hand, at 60°C we observe that the pellet with shell "3" does not perform as well as pellets obtained with shell "1 and 2" as it shows some weaknesses for the most severe storage conditions but without penalties on the storage duration time.

2.1. Asphalt mixture performances

During the asphalt laboratory fabrication, all GLOBE asphalt mixes have been made without any trouble mainly thanks to their aggregate cut-like size and form. The solid bitumen melted easily through heat transfer and mechanical stresses induced by mixing with hot aggregates. No extra time is required to obtain a visual satisfactory aggregates coating and homogeneous asphalt mix comparable to that of a pure bitumen introduced hot in liquid form. **Figures 6-a, -b and -c** exhibit the different characteristics of asphalt mix made with GLOBE solid bitumen in comparison with standard asphalt mix manufactured with 35/50 paving grade.

As shown in **Figure 6-b**, the use of GLOBE solid bitumen decreases the air void content in the asphalt mixture, that means that a better workability is achieved in comparison with asphalt mix based on common standard 35/50 paving grade. The workability is improved as the shell content ratio increases. For this class of asphalt mixture, the common specification for workability using Gyratory Compactor (PCG) is a void content between 5% and 10% at 60 gyrations. Asphalt mixture based on GLOBE solid bitumen also exhibit better rutting performances in comparison with 35/50 paving grade asphalt mixture as displayed in **Figure 6-a.** The rutting resistance seems to be slightly better as the shell content of GLOBE solid bitumen increases.

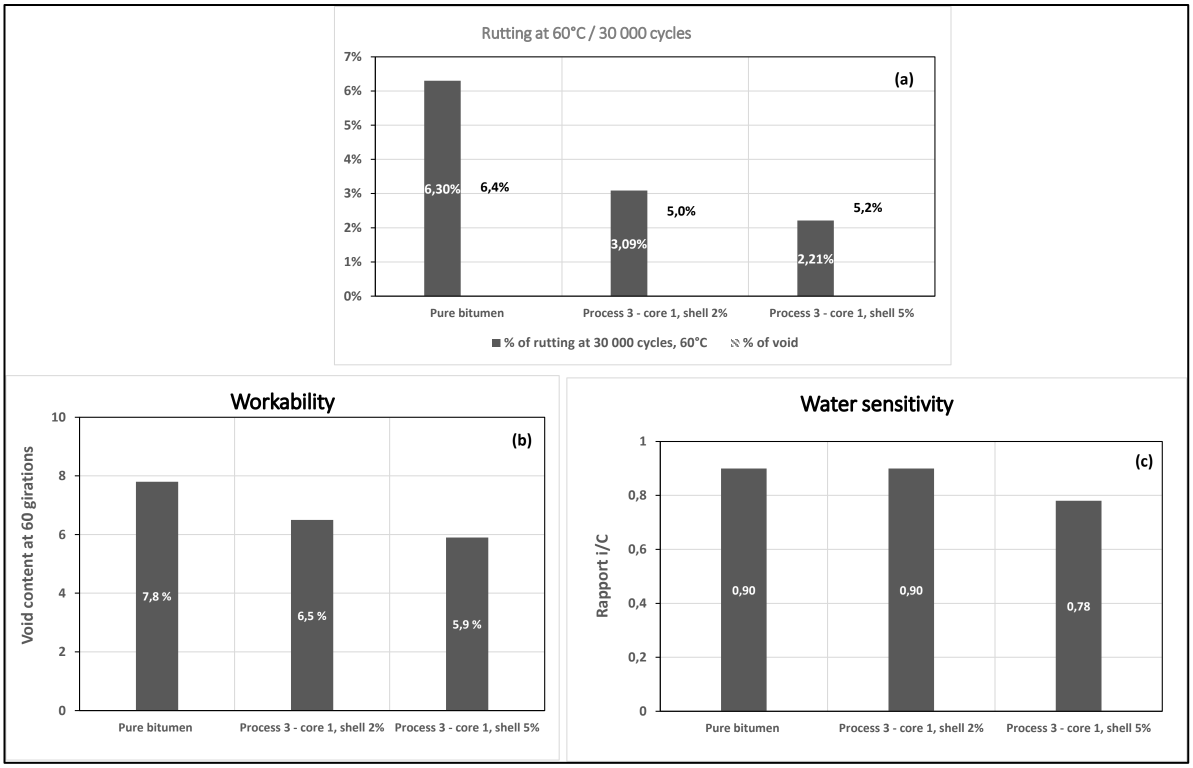


Figure 6 : Results of asphalt formulation study.

Regarding water debonding susceptibility, a 5% of shell content may induced a drop of I/C ratio (mechanical ratio between wetted specimen strength and dried specimen strength) in case of asphalt mixture made with GLOBE solid bitumen (**Figure 6-c**). However, at lower content of shell (2%), I/C ratio would be similar for GLOBE solid bitumen asphalt mixture and the 35/50 bitumen asphalt mixture.

Further investigations including life cycle assessment are needed to achieve a complete characterization and to fully assess the benefits of such sustainable innovation.

3. CONCLUSIONS AND PROSPECTS

Several technologies have been explored to design a GLOBE solid bitumen that would meet the defined expectations (temperature and loading conditions) for storage abilities. In the current state of the investigations, it appears that process "1" is the technology that gives the best results. The GLOBE bitumen obtained using well-chosen additives and proper coatings display the desired characteristics in terms of storage even under severe conditions of temperatures and loading stresses.

At asphalt mixture level, preliminary manufacturing through laboratory devices lead to satisfactory and homogeneous mixtures without any un-melted pellet residue. Pellets of similar to average aggregate size and form melted easily in contact with hot aggregates and under high mechanical shear stresses. It is expected that theses promising results would be even improved by upscaling in mixing plant thanks to bigger mechanical shear stresses.

In terms of asphalt mixture characteristic, current investigations made with GLOBE bitumen demonstrate similar or better performance regarding workability, rutting resistance. However, an excessive shell content would induce a drop of water debonding susceptibility and such items have to be part of further investigations.

Some challenges remain: extend the characterization of asphalt mixture including ageing and lifetime performances, RAP incorporation, plant manufacturing activities benefits, life cycle assessment and industrial risk benefits analysis along the whole road industry value chain to argue potential benefits/limitations for GLOBE solid bitumen ecosystem.

4. ACKNOWLEDGMENTS

The authors would like to thank the French Environment & Energy Management Agency"ADEME" for the co-funding support for the project. Special thanks to Romain Colliat, Jean-Philippe Paillac, Quentin Ambroisy, Frédéric Loup and Florent Shrevel for their contribution.

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Annex

Following to the preliminary asphalt mix study developed in the paper, this appendix presents a detailed evaluation of the impact of the use of Globe Bitumen pellets instead of conventional paving grade on the asphalt mix performances.

1. IMPACT OF GLOBE BITUMEN PELLETS ON THE MANUFACTURING OF ASPHALT MIX

In **Figure 1**, the scheme of manufacturing of asphalt mix is presented. It is observed that the use of Globe Bitumen pellets does not impact the manufacturing method, in particular:

- No extra heating of aggregates is needed. Globe Bitumen pellets melt easily in contact with the hot aggregates.
- No extra time of mixing is required to obtain a visual satisfactory aggregates coating and homogeneous asphalt mix.

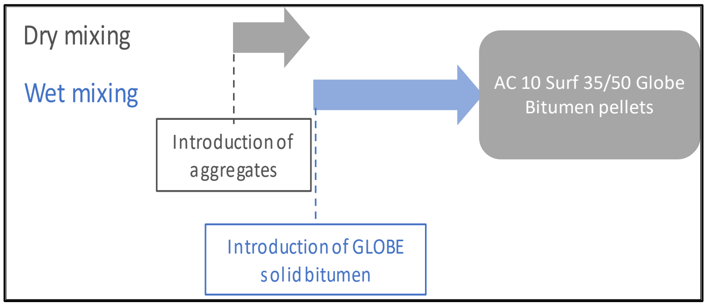

Figure 1 – Method of manufacturing

1.1. Comparison of Asphalt mixes performances between conventional 35/50 paving grade and GLOBE Bitumen pellets

In order to evaluate the impact of Globe Bitumen pellets on the performances of asphalt mixes., performances of conventional 35/50 paving grade and Globe Bitumen pellets 35/50 are compared in two different asphalt mixes : Surface layer (Asphaltic Concrete French mix design “BBSG3 0/10”) and Base course layer (Asphaltic concrete French mix design “GB3 0/14”). It is shown in **Figure 2**.

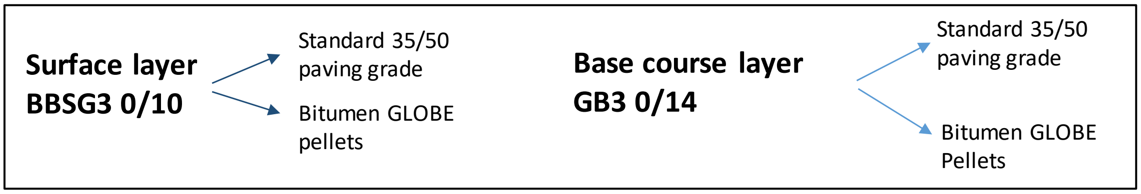

Figure 2 – Different asphalt mixes manufactured during the study.

1.2. Mixes characterizations

In order to investigate the effect of the shaping of bitumen on the asphalt mixes properties, several experiments are performed on mixes made either with the conventional bitumen or with the Globe one. In this appendix, only results obtained on surface layer asphalt mix are presented.

1.2.1. Workability

Experiments are performed according to the standard EN 12697-31. The void content at 60 gyrations is shown in **Table 1**. It is obvious that there is no impact of Globe Bitumen pellets on the workability of the asphalt mix. Indeed, the Percentage of void content in the two compositions are very close.

1.2.2. Moisture sensitivity

The test allows the determination of the water sensitivity of the bituminous mix. The compression resistances of the cores before and after being soaked in a thermostatic water bath are measured. Experiments are realized based on the standard EN 12697-12-B at 18°C. All results for Globe Bitumen pellets and conventional paving grade bitumen are shown in **Table 1**. *R* is the compression resistance of cores kept in air and *r* is the compression resistance of cores kept in water. Higher the ratio r/R is better is the water resistance of the mix. It is observed that both “in water” and “in air” compression resistance are close for an AC with Globe Bitumen pellets and for one with conventional bitumen grade.

1.2.3. Resistance to rutting

Rutting refers to permanent deformation of asphalt mixtures. The performance of the asphalt mixtures depends on the load frequency and on temperature, and also, there is a strong influence of the voids content.
Experiments are performed according to the standard EN 12697-22 (large size device) at 60°C, 1 Hz and results after 30000 cycles are shown in **Table 1**.

Table 1 – workability, Duriez and rutting resistance results for conventional and Globe Bitumen pellets.

	Conventional Bitumen 35/50	**Globe Bitumen Pellets**
Workability		
Void %	8.6	9.2
Duriez experiment (Mositure sensitivity)		
r (MPa)	8.4	8.4
R (MPa)	11.6	12.0
r/R	0.72	0.70
Resistance to rutting		
Void %	6.4	8
Depth at 3000 cycles (%)	3.9	2.7

First, using Gyratory Compactor we remark that the voids content is similar in both cases, showing a similar workability for all the bituminous mixes.
The depth at 60°C after 30000 cycles is lower for Globe Bitumen pellets than the one obtained with conventional bitumen. In fact, a better resistance to the permanent deformation is observed with Globe Bitumen pellets (2.7 vs. 3.9%) even if the percentage of void is higher (8% vs. 6.4% for conventional bitumen).

1.2.4. Stiffness and fatigue measurements

In order to look in more details at this comparison between Globe Bitumen pellets and conventional bitumen, Stiffness and fatigue measurements on hot mixes were performed based respectively on standard EN 12697-26 (Annex A) and EN 12697-24 (Annex A).

An increase of stiffness is observed with mixes made with Globe Bitumen pellets compared to mixes with conventional bitumen. On the other side, Fatigue resistance is quite similar for the two asphalt mixes. **Table 2** shows all the results obtained.

Table 2 – Modulus and fatigues results obtained.

	Conventional Bitumen 35/50	**Globe Bitumen Pellets**
Stifness Modulus at 15°C, 10 Hz, trapezoidal specimens		
Void %	6.4	6.7
Modulus (MPa)	10900	13500
Fatigue at 10°C, 25 Hz		
Void %	7.7	6.3
ε_6 µdef	97	105

2. CONCLUSIONS

In general, using Globe Bitumen pellets do not raise problem on the manufacturing of mixes and all characteristics and performances are unchanged or even sometimes slightly better.