Investigation of the rejuvenator content effect on the mechanical characteristics of cold recycled asphalt mixes

by

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Étude de l'effet de la teneur en rajeunissant sur les caractéristiques mécaniques des enrobés recyclés à froid

Bita EMAMGHOLI

RÉSUMÉ

Les revêtements d'enrobés perdent généralement leurs composants bitumineux essentiels avec le temps (à mesure qu'ils vieillissent), ce qui entraîne des fissures, une fragilité et, en général, de faibles propriétés mécaniques du revêtement. Par conséquent, les chaussées ont besoin d'un traitement partiel ou complet (recyclage) pour éviter leur détérioration. De nos jours, outre les méthodes classiques de recyclage à chaud, les technologies de recyclage à froid du fait de la limitation de la source d'énergie, mais aussi des préoccupations environnementales (élimination des émissions de fumées et de particules dans l'atmosphère) sont très demandées. Les activités d'entretien des anciennes chaussées (nécessitant une réhabilitation) à l'aide de matériaux secondaires et de déchets (granulats bitumineux recyclés (GBR) produits en concassant d'anciennes chaussées bitumineuses) sont des manifestations importantes de la part potentielle de la construction dans les objectifs de durabilité. Les enrobés recyclés à froid (CRM) peuvent être composés à 100 % de matériaux recyclés. Cependant, l'un des défis majeurs de ce processus est que le bitume inclus dans les matériaux recyclés n'est pas utilisable dans le nouveau mélange. Par conséquent, l'ajout d'une grande quantité de nouveau bitume à la chaussée de base augmente le coût du processus.

L'utilisation d'un rajeunisseur pourrait potentiellement mobiliser le liant vieilli inclus dans le matériau recyclé, et cela modifierait le comportement du mélange et du liant d'asphalte, ce qui entraînerait une plus grande quantité possible d'utilisation de GBR dans le mélange, une meilleure résistance à la fissuration et à l'orniérage. L'effet positif des régénérateurs dans le recyclage de l'asphalte à chaud a été approuvé dans la littérature. L'objectif principal de la présente recherche était d'évaluer la faisabilité d'utiliser un régénérateur avec des matériaux recyclés à froid. Les objectifs spécifiques étaient d'évaluer l'interaction physique et chimique entre le RAP et sa teneur en bitume avec l'additif rajeunissant. Cela visait à quantifier l'impact

de l'ajout de rajeunissant dans la réhabilitation des mélanges recyclés à froid (CRM) (comme la création de nouveaux clusters) et les propriétés rhéologiques du GBR (résistance à la traction), et ses propriétés correspondantes du bitume réhabilité. L'objectif secondaire était alors d'optimiser la quantité de bitume ajouté à ces enrobés en utilisant efficacement des régénérateurs dans différents scénarios de préparation d'enrobés à froid. Pour ce faire, l'effet de la teneur en rajeunissant dans différents aspects de la réhabilitation de l'asphalte, y compris l'effet de regroupement, et la caractérisation rhéologique des enrobés à froid ont été examinés dans différentes conditions de préparation des échantillons. Plusieurs caractéristiques d'échantillons et paramètres de préparation d'échantillons tels que la taille du size de GBR, la teneur en GBR dans le mélange, la température de préparation de l'échantillon, le temps de durcissement, le compactage ont été analysés en parallèle au pourcentage de teneur en rajeunissant dans le mélange. En outre, des tests FTIR ont été effectués pour déterminer si l'effet rajeunissant avait une nature de réaction chimique (nouvelle liaison chimique) ou s'il s'accompagnait d'une modification des propriétés physiques du mélange. Le GBR étudié a été collecté dans la région métropolitaine de Montréal, et le rajeunissant commercial SYLVAROAD PR1000 a été utilisé pour les investigations.

Les résultats ont indiqué que bien que la teneur en régénérateur puisse affecter de manière significative la formation d'agrégats dans l'enrobé à froid, la teneur en GBR et son size correspondant ainsi que le processus de préparation du mélange (compactage et température de préparation) avait un effet significatif à la fois sur la formation d'agrégats et les propriétés rhéologiques de l'enrobé. Dans le cas de la formation de nouveaux amas dans les matériaux réhabilités, les résultats ont montré qu'une augmentation de la teneur en rajeunissant entraînerait une augmentation de la formation d'amas. Cependant, cet effet était significativement accru lorsqu'il était combiné au compactage. Les mêmes tests IDT ont révélé que la teneur optimale en additif est significativement affectée par la température de préparation où une augmentation de la température de préparation de l'échantillon a entraîné une réduction de la teneur optimale requise en régénérateur (4 % pour la préparation de l'échantillon à 40 °C et 7 % pour la préparation à 60°C). De plus, l'analyse du bitume extrait a

montré qu'une augmentation de la teneur en rajeunissant dans le GBR initial pourrait augmenter les performances à basse et à haute température du bitume, cependant, le module complexe de cisaillement suggérait un pourcentage d'additif de 7 % comme teneur optimale dans cette configuration de matériaux. Enfin, l'analyse de la liaison chimique dans le test de spectroscopie infrarouge à transformée de Fourier (FTIR) n'a pu révéler aucune altération/production chimique efficace dans les groupes fonctionnels chimiques des matériaux testés à la suite de l'ajout de rajeunissant dans le bitume extrait réhabilité. En d'autres termes, les résultats suggèrent que les effets rajeunissants sont principalement contrôlés par la modification physique du mélange telle que la réduction de la viscosité, etc.).

Mots-clés : Rajeunisseur, GBR, Regroupement, Chimie du bitume, IDT, FTIR, Analyse rhéologique, Mélange à froid

Investigation of the rejuvenator content effect on the mechanical characteristics of cold recycled asphalt mixes

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ABSTRACT

Asphalt pavements usually lose their essential bituminous components by the time (as they age) which leads to cracking, brittleness and in general low mechanical properties of the pavement. Therefore, pavements need a partial or full treating (recycling) to prevent its deterioration. Nowadays, aside from the conventional hot recycling methods, cold recycling pavement technologies due to the energy source's limitation and also environmental concerns (eliminating of fume and particle emission to the atmosphere) are highly in demand. Maintenance activities of old pavements (that need rehabilitation) using secondary and waste materials (Reclaimed Asphalt Pavement (RAP) produced by crunching old bituminous pavement) is of important manifestations of the construction potential share in the sustainability goals. Cold Recycled Mixes (CRM) can be made of 100% of recycled materials. However, one of the major challenges in this process is that, the bitumen included in the recycled materials is not usable in the new mix. Therefore, adding a high amount of new bitumen to the base pavement increases the process cost.

The use of a rejuvenator could potentially mobilize the aged binder included in the recycled material, and it would change the behaviour of the mix and asphalt binder leading to higher possible amount of RAP usage in the mixture, enhanced cracking and rutting resistance. The positive effect of rejuvenators in the hot asphalt recycling has been approved in the literature. The main objective of the present research was to evaluate the feasibility of using a rejuvenator with cold recycled materials. The specific objectives were to assess the physical and chemical interaction between the RAP and its bitumen content with the additive rejuvenator. This was aimed to quantify the impact of the rejuvenator addition in cold recycled mixes (CRM) rehabilitation (such as new cluster creation) and RAP rheological properties (tensile strength), and its corresponding rehabilitated bitumen properties. The secondary

objective was then to optimize the amount of added bitumen to those mixes using effective employment of rejuvenators in different cold mix preparation scenarios. To do so the effect of rejuvenator content in different aspects of asphalt rehabilitation including clustering effect, and rheological characterization of the cold mixes were examined in different conditions of specimen preparation. Several specimen characteristics and specimen preparation parameters such as RAP size, RAP content in the mixture, specimen preparation temperature, curing time, compaction were analyzed in parallel to the rejuvenator content percentage in the mix. Besides, FTIR tests were conducted to find out the whether the rejuvenator effect had a chemical reaction (new chemical bonding) nature or it comes with some physical property modification in the mixture. The studied RAP was collected from the metropolitan area of the Montreal, and SYLVAROAD PR1000 commercial rejuvenator was used for the investigations.

Results indicated that although the rejuvenator content could significantly affect the clustering formation in the cold mix, the RAP content and its corresponding size as well as mixture preparation process (compaction and preparation temperature) had significant effect both on the cluster formation and rheological properties of the RAP such as tensile strength in IDT tests. In the case of new cluster formation in the rehabilitated materials, results showed that an increase in the rejuvenator content would result in an increase in the cluster formation. However, this effect was significantly increased when it was combined with the compaction. The same RAP IDT tests revealed that the optimum additive content is significantly affected by the preparation temperature where an increase in the specimen preparation temperature resulted in reduction in the required optimum content of rejuvenator (4% for specimen preparation at 40 °C and 7% for preparation at 60 °C). Furthermore, rehabilitated extracted bitumen analysis showed that an increase in the rejuvenator content in the initial RAP could increase the low temperature and high temperature performance of the bitumen, however, the shear complex modulus suggested 7% additive percentage as the optimum content in this setup of materials. Finally, the chemical bonding analysis in the Fourier Transform Infrared Spectroscopy (FTIR) test could not reveal any effective chemical alteration/production in chemical functional groups of the tested materials as a result of rejuvenator addition in the

rehabilitated extracted bitumen. In other words, results suggest that the rejuvenator effects are mostly controlled by the physical modification of the mix such as viscosity reduction, etc.).

Keywords: Rejuvenator, Reclaimed Asphalt Pavement (RAP), RAP clustering, bitumen chemistry, IDT, FTIR, Rheological analysis, Cold mix.

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LIST OF ABBREVIATIONS

AC	Asphalt Concrete
CM	Cold Mixture
CIR	Cold In-place Recycling
HIR	Hot In-place Recycling
CRM	Cold Recycling Mixture
CR	Cold Recycling
CTM	Cement Treated Material
FDR	Full-Depth Reclamation
HMA	Hot Mix Asphalt
RA	Reclaimed Asphalt
RAP	Reclaimed Asphalt Pavement
SGC	Shear Gyratory Compactor
Agg	Virgin Aggregate
Rej/REJ	Rejuvenator
DSR	Dynamic Shear Rheometer
BBR	Bending Beam Rheometer
FTIR	Fourier Transform Infrared Spectroscopy
IDT	Indirect Tensile Test
ST	Stiffness
ΔT_C	LTs-effective – LTm-effective
LT_s	Low temperature (driven from stiffness curve)
LT_{m-v}	Low temperature (driven from m-v curve)
LT	Minimum size number between LT_s and LT_{m-v}
LT_e	Low temperature effective (resistant against first crack)

INTRODUCTION

In this era, construction sectors play an important role in the world economy. Construction is one of the major consumers of raw materials, chemicals, electrical and electronic equipment, etc. (Principi, Catalino, & Fioretti, 2015). Besides, the energy performance of constructions and usage of materials of structures and infrastructures have a significant influence on energy, climate changes and environment. Therefore, it can be said that the construction and sustainability have a very strong relationship. In other words, optimized and environmentally friendly construction and infrastructures have a strong capacity to take participate in a more sustainable future of the Earth. Sustainable construction can incorporate different elements of economic efficiency, environmental performance and social responsibilities or, in other words, *The Three Pillars of Sustainability*. It should be noted that no system is sustainable as long as all three pillars of sustainability are not satisfied.

Nowadays, major social and economic policies as well as new national and continental guidelines are moving forward to achieve sustainability goals, such as reusing of secondary and waste materials. Specifically, road infrastructures designing, and maintenance activities of old pavements (which need rehabilitation) are important manifestations of the construction potential share in the sustainability. Roads have always been an inseparable part of the civilized world and date back to more than 4000 years B.C. Development and roads necessitate each other in the modern world. Humankind started to find new habitats and to do so, roads seemed essential. Today, there are thousands of kilometres of roads all over the world including Canada. In Canada, there are 1,042,300 kilometres of roads so far and this number is an ever-increasing figure due to population growth, and advances in equipment and knowledge makes the development happen in a faster way than before. During the last 50 to 70 years, population growth and economic progress provided extensive network of asphalt paved roadways. The road industry, as a big material and oil consumer, has been affected by sustainability enormously. New equipment and new executive approaches emerged as the outcomes of many years' efforts revolving around sustainability. Material specification, hauling and type beside design protocols and standards have been undergoing lots of changes and revisions. Several parameters like road layout (e.g., size, length, location, landscape impact) and pavement structure (e.g., layer thickness, materials, manufacturing techniques) should be taken into consideration to have a sustainable road construction.

Therefore, any solution or method should simultaneously satisfy environmental, social and economic concerns. For instance, aggregates used in construction should comply with all the requirements of predefined sustainability standards (concerning the recycled materials, stability of the materials, etc.) as well as the structural requirements to have desirable performance. Bituminous layers, as the very first layer of the road which touches tensions, has a crucial role to play in a matter of performance and durability of the useful life of a road section. A desirable performance of a flexible pavement is a result of a synergy between the bituminous
layer and sub layers. As it comes to the rareness of qualified raw material, reusing the existing old pavement particles seem to be the only solution. Reclaimed Asphalt Pavement (RAP) comes from milling or scraping old pavement and after processing, it can be used in a new pavement. Using recycled materials reducing the demand for extraction can provide more economic investment on the road construction.

Background (asphalt recycling)

Asphalt pavements usually lose their essential bituminous components by the time (as they age) which leads to cracking, brittleness and in general low mechanical properties of the pavement. Therefore, pavements need a partial or full treating (recycling) to prevent its deterioration. Besides, new demands like enhancing the user's riding comfort, road safety and finally as previously mentioned economic limitations and environmental concerns provided a situation in which rehabilitation overcome the new constructions in many countries like

Canada. One of the most important and trended ways to increase the effectiveness of existing budgets (materials in the existing pavement) in order to maintain, rehabilitate and reconstruct more kilometres of roads for each spent dollar is asphalt recycling (Manual, 2001). Asphalt recycling specifically during the last two decades due to a big increase of the oil price along with limitations in accessibility of aggregates with desirable quality got more importance.

There are several advantages for recycling including (Oke, 2011; Xiao et al., 2018): cost effectiveness, environmental impact, fuel and raw material consumption, safety, easiness of the application and potential pavement improvements, elimination of a disposal problem. Different types of asphalt recycling methods are in practice all over the world. Generally, all methods can be narrowed down to three well-known methods of asphalt recycling including:

- Hot In-place Recycling (HIR)
- Cold In-place Recycling (CIR)
- Full-Depth Reclamation (FDR)

These methods are applicable to different types, levels, and severity of the distresses, and consequently different periods of the pavement life. CIR with bitumen needs relatively lower amount of energy in comparison to the conventional methods such as HIR. Cold Recycled Mixes (CRM) can be made of 100% of recycled materials. However, one of the major challenges in this process is that the bitumen included in the recycled materials is not usable in the new mix (because it is aged). Therefore, during the asphalt pavement creation new virgin bitumen should be added to the materials to be used as binder between aggregate and RAP. adding a high amount of new bitumen to the base pavement increases the process cost. Adding excessive new virgin bitumen eliminates the advantage of recycling and therefore a solution to minimize this should be found. Although the reasons to justify the use of cold recycling techniques are adequately addressed in the industry and in the literature, some barriers still prevent them from development and employment in many cases including lack of sound standards for the mix design and its corresponding application, deficient understanding about the behaviour and failure mechanism of cold mixes, lack of knowledge on the new additive effect on the improvement of the cold recycling effectiveness, etc. This results in a situation in which cold recycling methods are mainly employed based on empirical correlations and developed best practice based on the experience (K. J. Jenkins, 2000). However, promisingly, a lot of research recently conducted and ongoing researches aim to determine the specification and behaviour of Cold Recycling mixtures to fill the gap of knowledge in this field (Apeagyei & Diefenderfer, 2012; Kim et al., 2010; Stimilli, Ferrotti, Graziani, & Canestrari, 2013). One of the key elements in the field of cold recycling improvement with the objective of less raw materials consumption is the field of additives. Several additives have been investigated in the literature (such as lime, cement). Recently, rejuvenators have raised significant interest among researchers to increase the effectiveness of pavement recycling. The use of a rejuvenator could potentially mobilize the aged binder included in the recycled material, and it would change the behaviour of the mix and asphalt binder leading to higher possible amount of RAP usage in the mixture, enhanced cracking and rutting resistance. The positive effect of rejuvenators in the hot asphalt recycling has been approved in the literature. However, there is little information on the effect of rejuvenators on the cold recycling methods.

Research Problem

There are several parameters in the field of cold recycling and rejuvenators employment to increase the asphalt payments rheological performance. Several criteria which influence the final cold mixtures' properties such as RAP sources, manufacturing process, bitumen types, asphalt sample preparation condition like compaction and preheated specimen temperature, different volumetric properties and so forth. Although, in the last two decades the knowledge about the cold mixes is improved considerably, but there are still new grounds to break on CR, specifically in the optimization of the mix design and production process which in turn needs comprehensive study on the parameters and subsequent experimentation to find out the optimum conditions. The rejuvenator addition to the cold mixes to rehabilitate aged bitumen performance is one of the important areas in this filed which remarkably affects the final characteristics of the mix. However, the absence of comprehensive knowledge makes it to be an interesting topic for lots of research attempts to find out the effect and subsequently optimize its usage in the field pavements. Although, there is some information about the effect of rejuvenator usage on the hot recycling method, little information about the effect of rejuvenator usage and their impact on the cold recycling mixes is available in the literature. Therefore, the problem is we do not precisely understand the impact of rejuvenators on the performance improvement of cold recycling mixes and its ingredients including RAP and its bitumen content. This includes both direct impact and cross functional impact when it comes to investigation of rejuvenator's impact in different mix preparation protocols (i.e., temperature, compaction, RAP size, etc.). Finally, yet importantly it needs to be clarified whether the rejuvenators impact origins in the chemical reactions between rejuvenators and aged bitumen or it has roots in the physical impacts (such as reduction in effective viscosity).

Research Objectives

This study attempts to analyze the effect of rejuvenators on the performance of cold recycling mixes. It's an endeavour to determine the origins of the rejuvenator interaction with cold mixes ingredients (including RAP and its bitumen content) which would probably provide us with a more realistic overview about it and would be helpful in matter of behavioural prediction. Finding an optimum amount of rejuvenator in the cold mixes to increase the pavement rheological performance as well as identification of rejuvenator role's nature (chemical or physical) and its possible chemical reactions with the aged bitumen are the research motivations of the present study. Therefore, two minor objectives were to identify the effect of rejuvenators on a) RAP and b) bitumen content in the cold mixes.

To do so following minor objectives were tracked during the research different phases:

- Investigating the rejuvenator impact on the cluster creation in RAP, considering RAP source parameters' (i.e size) cross functional impact on the final cluster formation which leads to a more comprehensive understanding about it.
- Investigating the rejuvenator effect on the rheological properties of cold mixes, considering different mix preparation protocols including RAP content, compaction and temperature of mixture
- Determination of the rejuvenator usage on the rehabilitation and performance of aged bitumen content of RAP.
- Tracking the chemical interaction of rejuvenators with the bitumen content of cold mixes to drive awareness and knowledge about the physical or chemical origin of the rejuvenator's role in the filed of cold asphalt recycling.

Organization of the Thesis

This present thesis is arranged in three chapters along with this introduction which start with the problem statement and objective of the thesis in this first chapter. In this introduction, thesis tries to start a discussion through roads, asphalt pavement and their rehabilitation. Afterward, it introduces the recycling concept and different techniques for rehabilitation, and it is followed by the problem statement and objective of the thesis. In the first chapter, a concise and complete literature review about cold recycling, cold mixtures and definitions about different kinds of them are presented. Cold mixture ingredients and the role they play in the mix are also included. Besides, after introduction of rejuvenators, the effect of rejuvenators on the cold recycling processes on the literature and available information in this regard are discussed. The second chapter is a full description of the material and methods that are used in the laboratory, and different protocols and processes that have been exercised. It includes details of different test processes that are used to evaluate the rheological performance of the cold mixes as well as physical and chemical evaluation tests that are conducted to identify the effect of rejuvenators on the cold mixes in this study. In chapter three, the results of the tests are fully presented. This chapter includes result analyzes and discussion in each section to drive conclusions about the role of rejuvenators on the cold mixes performance and suggest some optimized content in this setup of materials. Conclusion following with some suggestions for the continuation of this work are the final part of the thesis.

CHAPTER 1

LITERATURE REVIEW

This part of the thesis mainly covers the related literature review of the research. First, the need for the sustainable road construction and asphalt recycling discussed and then the components of such process are illustrated in detail. Different ingredients of the cold mixes including the additives and rejuvenators as the target ingredient of the present research are introduced. Chapters continue with detail information about the state of the art on the effect of rejuvenator usage on the rheological properties of the cold mixes. Finally, chapter closes with a summary and elaboration on the research objectives according to the state of the literature.

1.1 Sustainable Road Construction

Nowadays, major social and economic policies as well as new national and continental guidelines are moving forward to achieve sustainability goals, such as reusing of secondary and waste materials. Specifically, road infrastructure designing, and maintenance activities of old pavements (which need rehabilitation) are the important manifestations of the construction potential share in the sustainability. Using recycled materials reducing the demand for extraction can provide more economic investment on the road construction. Current research and practice tend to focus on the use of waste and recyclable materials in the lower courses of the road (base, subbase, etc.) during the road construction, because they absorb materials in larger quantities in comparison to the upper courses.

However, aside from the initial cost of construction, as the roadway network expands and traffic volume augmented, and simultaneously budgets tightened increased highlighting has been concentrated on maintenance and preservation of the existing roadways and their pavements. In different countries like European countries or cold area like Quebec, highway authorities are mainly dealing with the maintenance and repairing the roads rather than its new construction. Repairing of a road affects both upper and lower pavement layers of the road structure on the basis of its condition. Therefore, the demand for comprehensive studies to prove the possibilities of new technologies in road recycling is very vital from the sustainable point of view. Besides, the optimum time to have the maximum performance should also be investigated (See figure 1.1). According to the Waste Framework of Directive (Principi et al., 2015) the final goal of 70% usage of recycling materials in construction by 2020 represents the economic value of this development.

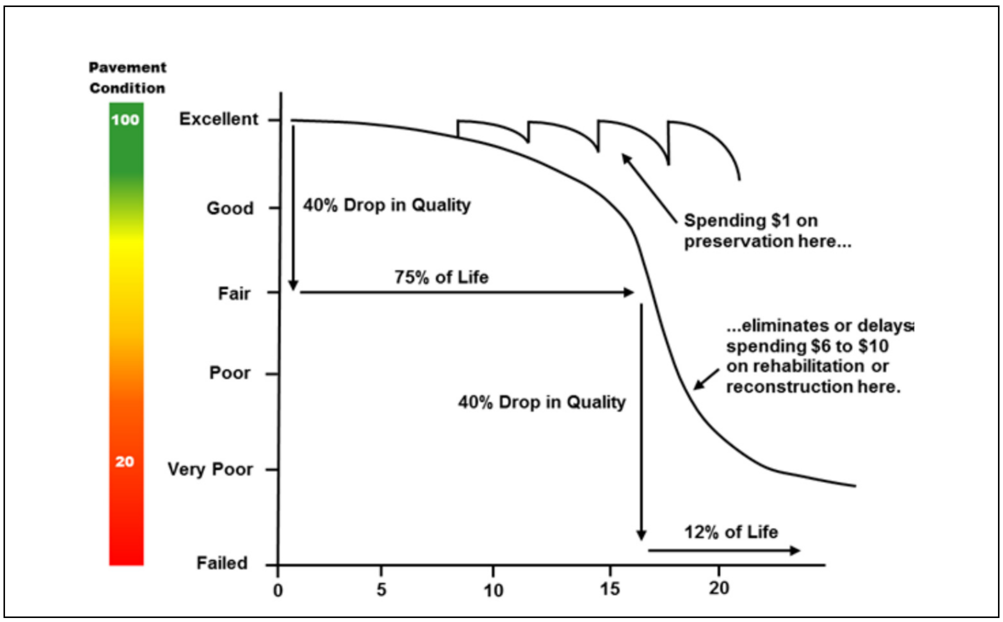


Figure 1.1 Pavement rehabilitation and its relative time and quality
Taken from Godenzoni (2017)

There are several advantages for recycling including (Oke, 2011; Xiao et al., 2018) including cost effectiveness, environmental impact fuel and raw material consumption, safety, easiness of the application and potential pavement improvements, elimination of a disposal problem. These methods (HIR, CIR, FDR) are applicable to different types, levels, and severity of the distresses, and consequently different periods of the pavement life (Figure 1.2). In general, HIR is applicable to the roads where the majority of the pavement distresses are minimal and limited to the upper layer of the pavement surface with no trace of structural problems like longitudinal cracking in the wheel path, alligator or edge cracking. On the other hand, CIR is applicable where higher number and severity of distress exist not only on the surface, but also down from the surface. FDR can be used for reconstruction, profile improvement and structural capacity development. Nowadays, an extensive different hot recycling methods is common practice and this method is still the most common way of recycling in Europe (Karlsson & Isacsson, 2006). However, during the last decade cold recycling proving its highest potential in terms of energy saving and emission reduction, attracted lots of interest in different countries like Canada.

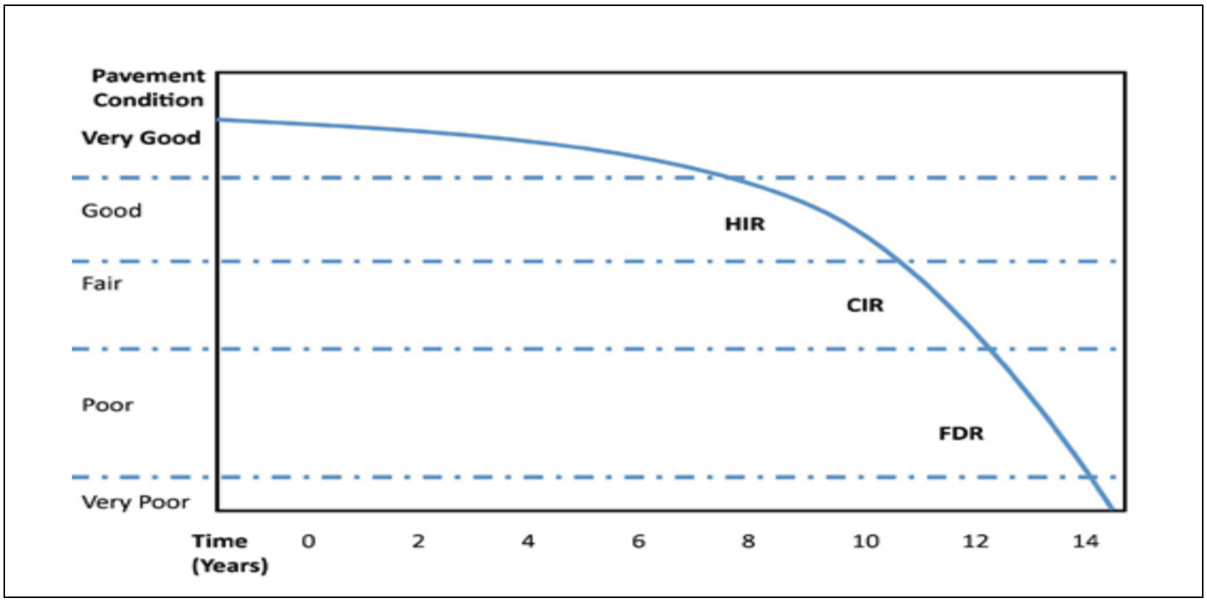


Figure 1.2 Different in-place recycling method based on pavement condition
Taken from Godenzoni (2017).

Figure 1.3 shows comparative graphs of energy usage in different asphalt mixes. It is clear that the CIR with bitumen needs relatively lower amount of energy in comparison to the conventional methods such as HIR. In the following chapters, cold recycling method and its relevant parameters and evaluation standards are presented in detail.

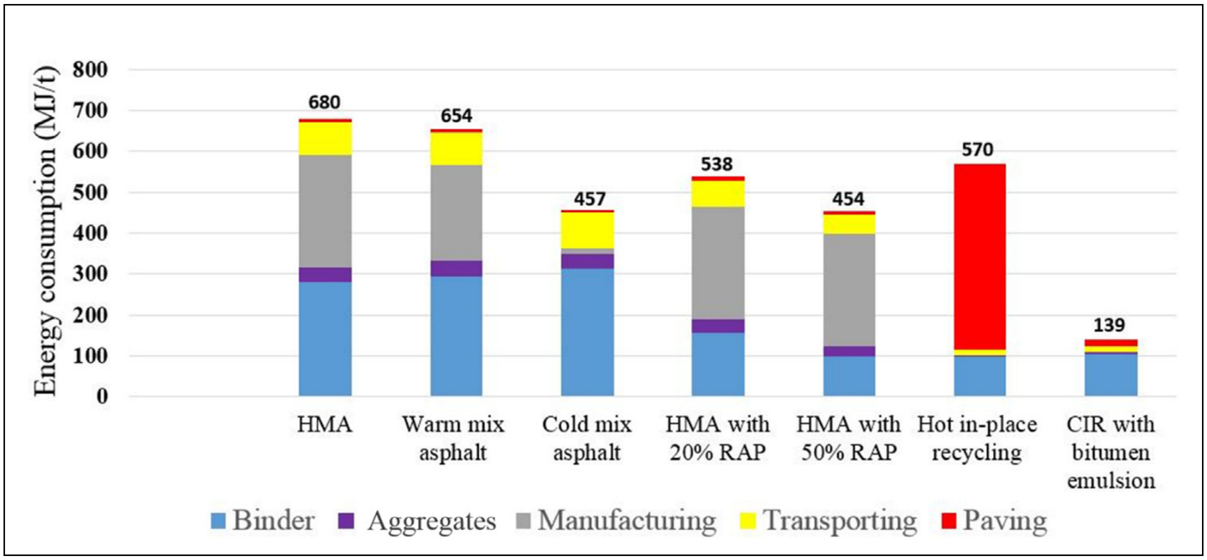


Figure 1.3 Energy consumption of different asphalt products

Taken from Chehovits and Galehouse (2010).

1.2 Cold recycling

In today's world of asphalt pavement technology, new techniques, technologies and apparatus are employed to mill the pavement at different depths and add various binder to the recycled materials like bitumen emulsion or foamed bitumen. Different additives like lime and cement are also considered during the recycling to further improve the performance of the pavement. However, this technology roots back to the 1900s where the asphalt recycling was increasingly used in developed countries (A. Recycling, Association, & Dunn, 2001). The cost of equipment, lack of reliable guidelines for mix design as well as incomplete understating about the failure behaviour and different parameters contributed in the performance improvement of the cold recycling resulted in limitations of this application into low traffic roads (on the contrary to the HMA method) (Apeagyei & Diefenderfer, 2013; TODD & Karmas, 2003). Cold recycling generally goes through two different methods, namely in-plant and in-place. In the former method, the recovered material is hauled to a depot plant and used in the mixing process, while, in the latter method all the process takes place in once at the place of recycling and in the recycling machine (C. Recycling, 2010). Although, the in-place is cheaper and more environmentally friendly due to the elimination of hauling the control on different ingredients and their contributing parameters is more prominent in the in-plant method (Xiao, Yao, Wang, Li, & Amirkhanian, 2018).

1.3 Cold mixtures

In current practices, cold mixtures usually consist of an aggregate blend (composed of virgin or reclaimed aggregates), bitumen in form of bitumen emulsion or foamed bitumen, cement and sometimes, other additional stabilizing agents such as lime or fly ash. Water is used to allow cold mixture lay-down and compaction. It should be mentioned that the simultaneous presence of bitumen and other active or inactive fillers (e.g., cement, calcium carbonate, hydrated lime, etc.) do not produce a new binder in the mixture. Addition of binding agents serves to have suitable structure and durable properties (Bocci, Grilli, Cardone, & Graziani, 2011).

Aggregates are produced in quarries in the usable size of 50 mm to less than 0.075 mm, and they have mechanical properties inherent from its parent rocks (Mallick & El-Korchi, 2008). Therefore, based on their geologic origins, they should be evaluated for the cold mixtures to assess several properties like their suitability for bituminous mixtures, durability, resistance to cold weather and breakdown under freeze-thaw. The main goal of aggregate blend design is not only to remain as much as possible to the maximum density gradation in order to avoid too high voids and provide stable structures, but also to maintain enough space for bitumen binder. Recycled materials like Reclaimed Asphalt Pavement (RAP) have been extensively used in asphalt mixtures owing to their environmental, economic, and social benefits (Hansen & Copeland, 2015). Aside from the European countries like Switzerland, American asphalt designers and producers in different US states (according to the National Asphalt Pavement Association (NAPA)) effectively use RAP as a component of new mixtures ((see Figure 1.4). Generally, when RAP is replacing virgin aggregate in cold mixture, the mixture is referred as cold recycled mixture (CRM).

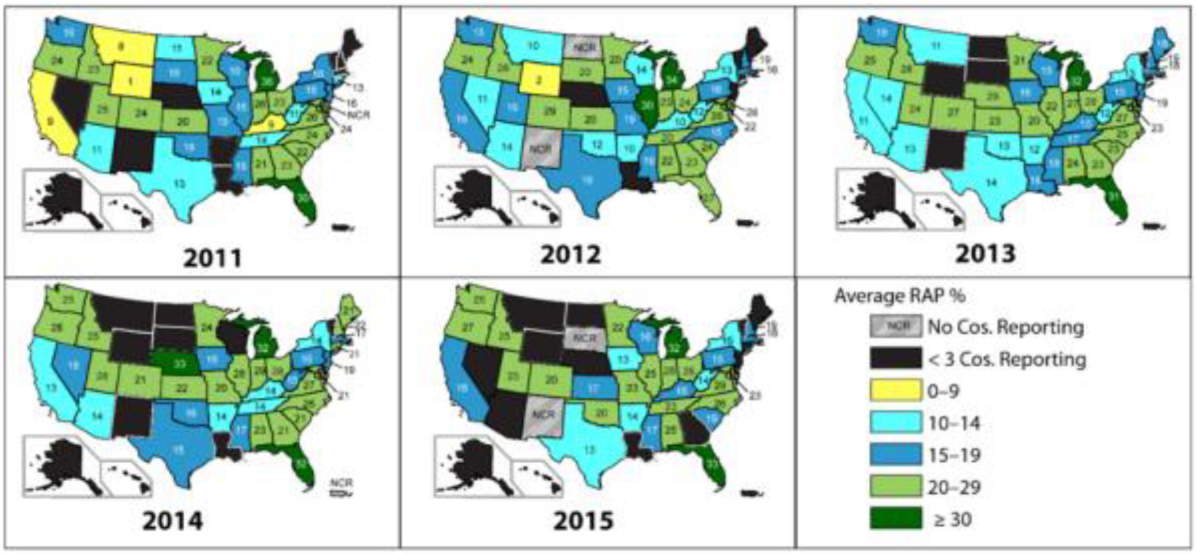


Figure 1.4 The schematic presentation of average RAP used in asphalt mixtures in US states Taken from Hansen and Copeland (2015).

1.4 Reclaimed asphalt pavement (RAP),

RAP obtained from removed asphalt pavements in road networks or small sites like parking lots, plant waste, or rejected paving mixture (which should be subjected to further process before reusing) is the most widely used recycled material in new mixtures. It is considered as a source of recycled aggregate and asphalt binder for the new mixtures. Cold material is a multiphase material consisting of: Solid particles (virgin or reclaimed aggregates, etc. in the cured state of the mixture), fluids (like water and bitumen emulsion in the fresh state of the mixture) and finally air voids (see Figure 1.5).

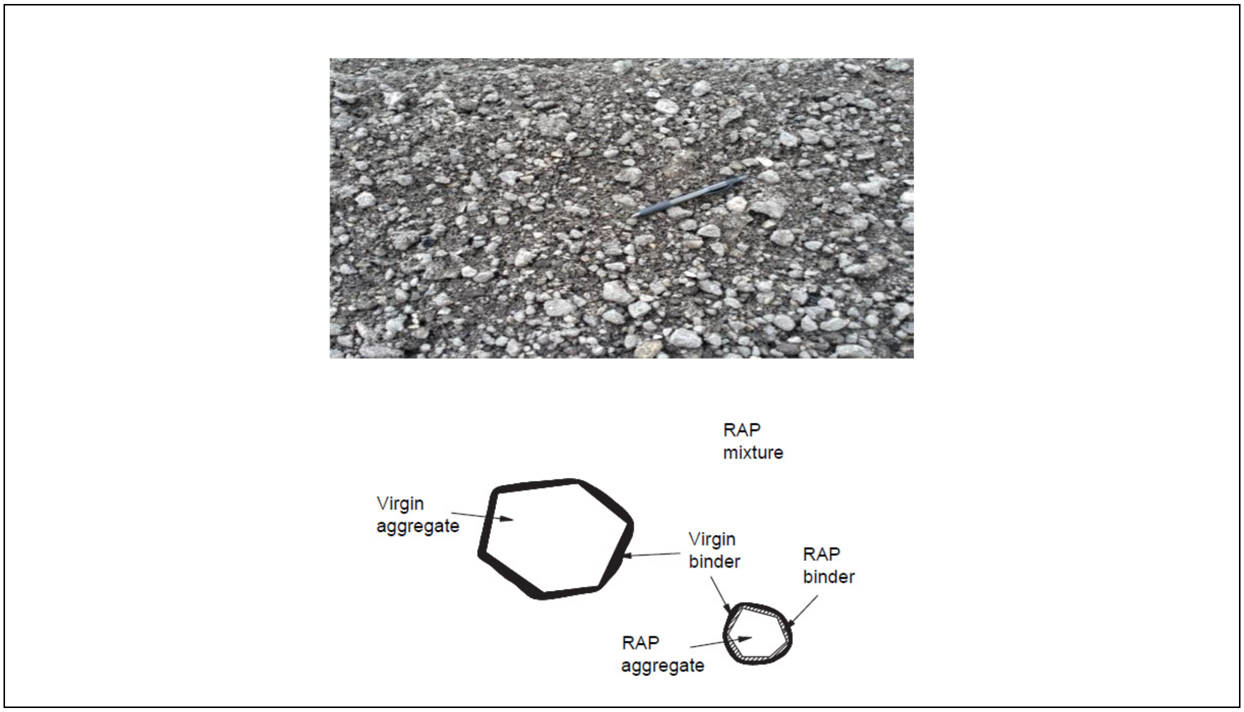


Figure 1.5. Reclaimed asphalt pavement and its schematic material mixture (RA)
Taken from Huang, Li, Vukosavljevic, Shu, and Egan (2005).

Cold mixtures can be categorized in different ways. Based on their gradation, they can also be organized as dense graded, sand and open graded (Huang, Li, Vukosavljevic, Shu, & Egan, 2005). One of the common practice to classify the cold mixes is to arrange them based on type and dosage of binding agents (bitumen and cement) as follows (Godenzoni, 2017) (See also Figure 1.6):

- Cement-treated materials (CTMs): Conventional cement-treated materials are usually used in the base course to improve the bearing capacity of pavements and distribution of traffic-induced stresses. Recycled aggregates such as crushed concrete, crushed masonry and reclaimed aggregates are some can be used in CTMs. They usually have high stiffness and brittle behaviour.
- Bitumen-stabilized materials (BSMs): This category, using bitumen emulsion or foamed bitumen as binding agent, is very common in practice. They have following characteristics:

1) significant improvement in the cohesion, 2) Flexural strength, 3) improved moisture sensitivity and lower tendency to pump under loading.

- Cement-bitumen treated materials (CBTMs): CBTMs are usually used as subbase material and characterized by higher cohesion and stiffness in comparison to BSM. In fact, starting from CTM and stepwise adding of bitumen to reduce the vulnerability to cracking is the main idea if the CBTMs production procedure.
- Cold-mix Asphalt (CMA): Cold mix asphalt (like G-E (Raschia, 2020)) using bitumen emulsion or foamed bitumen as bituminous binding agent are getting used as base course strengthening old pavements. CMA is designed to have minimum vulnerability to rutting and good partial coating of aggregates. CMA is usually sensitive to water and should be used with surface drainage. Generally, CMA should be compacted by heavy pneumatic rollers to improve internal friction and cohesion.

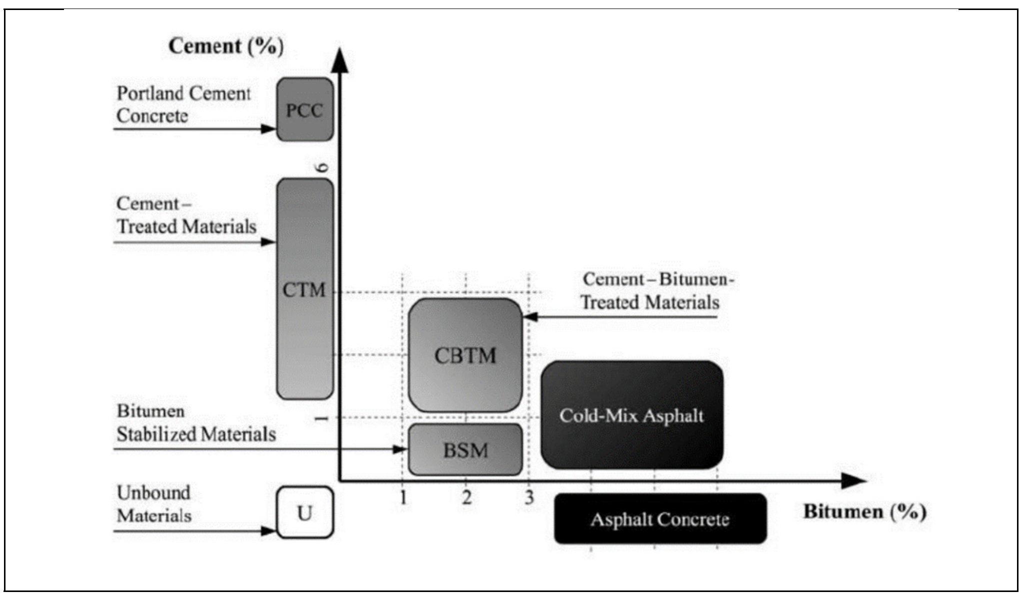


Figure 1.6 Different pavement mixture compositions
Taken from Godenzoni (2017).

1.5 Bitumen

1.5.1 Definition

Bitumen is defined by Chambers and Ephraim (Chambers & Rees, 1784) in their early modern dictionary as a “generic term encompassing naphtha, petroleum, pitch and most mineral hydrocarbons forms, whether hard, soft or liquid”. It is the viscous mostly-hydrocarbon component of natural asphalt and can be considered an extra-heavy oil (Lesueur, 2009). It can also be defined in an extended way as viscous petroleum distillates containing a low percentage of volatile compounds. More generally, it can be defined as a virtually in volatile adhesive and waterproofing material derived from crude petroleum which is very viscous or (even nearly solid) at room temperature (Lesueur, 2009).

1.5.2 Bitumen physical and chemical properties

Typically, bitumen density varies between 1.01 and 1.04 g/cm3 depending on the crude source and paving grade. The bitumen chemistry is very complex due to the fact that the many different materials are present in it. However, a general description of the chemical nature of crude oil can be separated as paraffinic, naphthenic or aromatic if a majority of saturate, cyclic or aromatic structures, respectively, are present (Lesueur, 2009). Table 2.1 provides an overview of bitumen elemental analysis in different regions including Canada as the case of this study.

Table 1.1 Elemental analysis of bitumen

Taken from Lesueur (2009).

Origin		AAA-1 Canada	AAB-1 USA	AAC-1 Canada	AAD-1 USA	AAF-1 USA	AAG-1 USA	AAK-1 Venezuela	AAM-1 USA
C	wt.%	83.9	82.3	86.5	81.6	84.5	85.6	83.7	86.8
H	wt.%	10.0	10.6	11.3	10.8	10.4	10.5	10.2	11.2
H+C	wt.%	93.9	92.9	97.8	92.4	94.9	96.1	93.9	98.0
H/C	Molar	1.43	1.55	1.57	1.59	1.48	1.47	1.46	1.55
O	wt.%	0.6	0.8	0.9	0.9	1.1	1.1	0.8	0.5
N	wt.%	0.5	0.5	0.7	0.8	0.6	1.1	0.7	0.6
S	wt.%	5.5	4.7	1.9	6.9	3.4	1.3	6.4	1.2
V	ppm	174	220	146	310	87	37	1480	58
Ni	ppm	86	56	63	145	35	95	142	36
Mn	g/mol	790	840	870	700	840	710	860	1300

As it can be seen the elemental composition of bitumen mainly depends on its crude source rather than its geographical generalization. For example, various materials from the USA and Canada being in the same region have quite different composition. In general, bitumen mainly consists of carbon (80-88 wt%), hydrogen atoms (8-12 wt%) or, in other words, more than 90% of hydrocarbon content. Other materials, like sulphur, nitrogen and oxygen are generally in the bitumen. Functional groups as combinations of these materials are presented in Figure 1.7.

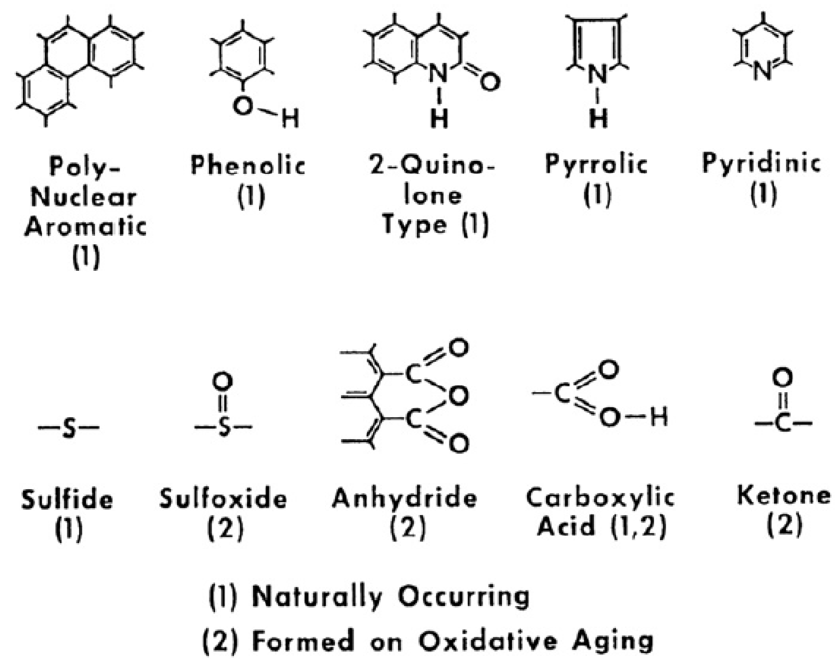


Figure 1.7 Functional groups present in bitumen
Taken from Lesueur (2009).

The separation of bitumen into its various functions is presented in Figure 1.8, which is usually highlighted as SARA fractions. In this section, each function is discussed briefly. ***Saturates*** usually are 5-15 wt% of paving grade bitumen. They produce a colourless or slightly coloured liquid at ambient temperature. This is because of a very low glass transition temperature (around -70º C) which is normally 40 degrees below than their parent bitumen glass transition. Their density is around 0.9 g/cm^3 at room temperature while, they possess a solubility between 15 and 17 MPa(Lesueur & Potti, 2004).

Aromatics which are usually called as naphthalene aromatic can be considered as one of the most plentiful fractions of bitumen which usually form a yellow or red liquid at room temperature and has more density and solubility in comparison to the saturates (inferior to 1 g/cm^3 and 17 to 18.5 MPa). They with resins consist about 30-45 wt. % of the total bitumen at room temperature. Aromatic carbon skeleton is slightly aliphatic with lightly condensed aromatic rings.

Resins or polar aromatics, which are black solids at room temperature, play a critical role at bitumen stability due to their stabilizer act for the asphaltenes. They have a density of around 1.07 g/cm^3 and solubility of 18.5-20 MPa.
Asphaltenes providing 5-20 wt.% of a paving grade bitumen represent low solubility class of bitumen molecules which has about 1.15 g/cm^3 of density. They form a black powder at room temperature are generally responsible for the black colour of the bitumen.

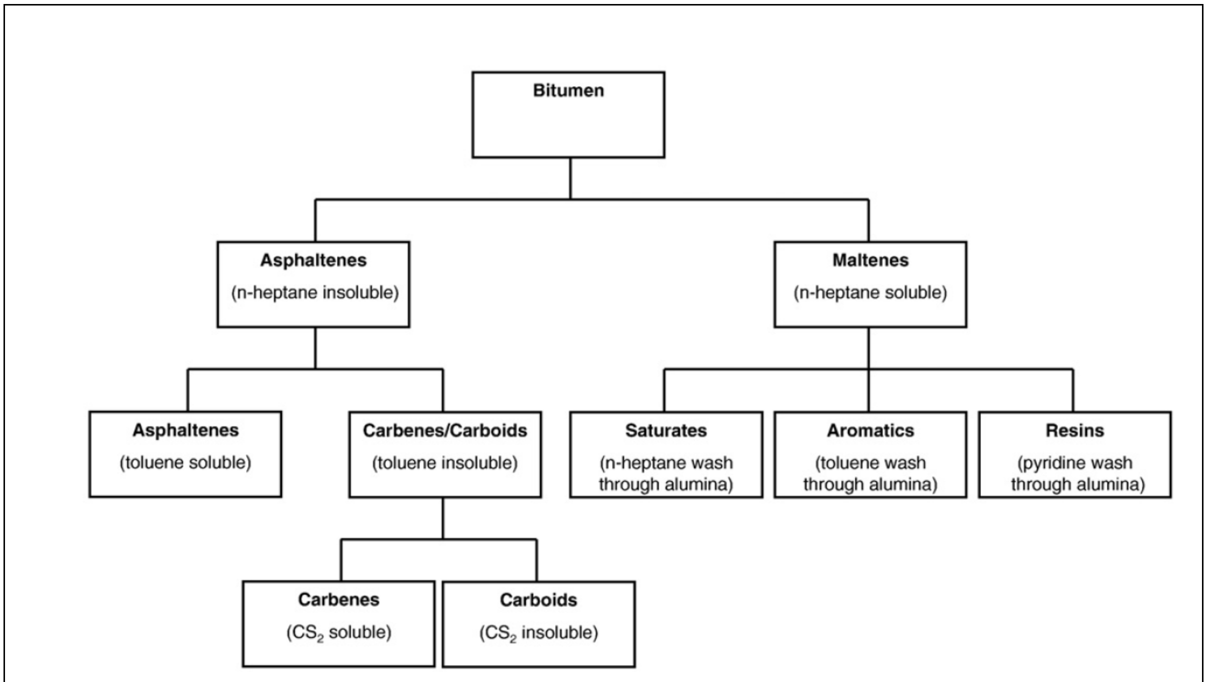


Figure 1.8. Schematic flowchart of bitumen separation into its fractions
Taken from Lesueur and Potti (2004).

1.6 Cold mix materials important properties

There are some important properties of cold mix materials, which should be considered to not only analyze their behaviour, but also evaluate their performance as follows:

- **Workability**: The ability of asphalt to lay down which should be controlled through the breaking kinetics. This property is not measured directly and is usually identified using indicators like air void content in the mixture. However, for the cold mixes Nynas workability test is recommended.

- **Compatibility**: This parameter in the cold mix design evaluation is measured directly. It is usually expected that the well-compacted asphalt has about 15 % of air void in the texture of the mix. Marshal compaction method and gyratory compaction are two well-known methods to compact and evaluate the compatibility of the cold mixes. However, there are lots of uncertainties about compaction methods and yet it needs to be cautiously evaluated through the asphalt making.
- **Mechanical properties**: These properties in the case of cold mixes should be evaluated through time. In other words, these properties are time-dependent properties in this field of study. This is mainly because of the fact that the breaking and water evaporation are time-dependent processes. Direct measurements of tensile test, fatigue test, compaction resistance test, modulus test are some of the well-known methods to evaluate the mechanical properties of the mixes.

Cold materials as like as traditional asphalt concrete have a viscoelastic behaviour. Showing a time-temperature dependent behavior they change in the exhibited properties by the load frequency and load path. This behaviour is strongly correlated with the volumetric properties of the component materials. A typical mix behaviour domain as a function of applied cyclic loading (N) and strain amplitude (ε) as depicted in Figure 1.9.

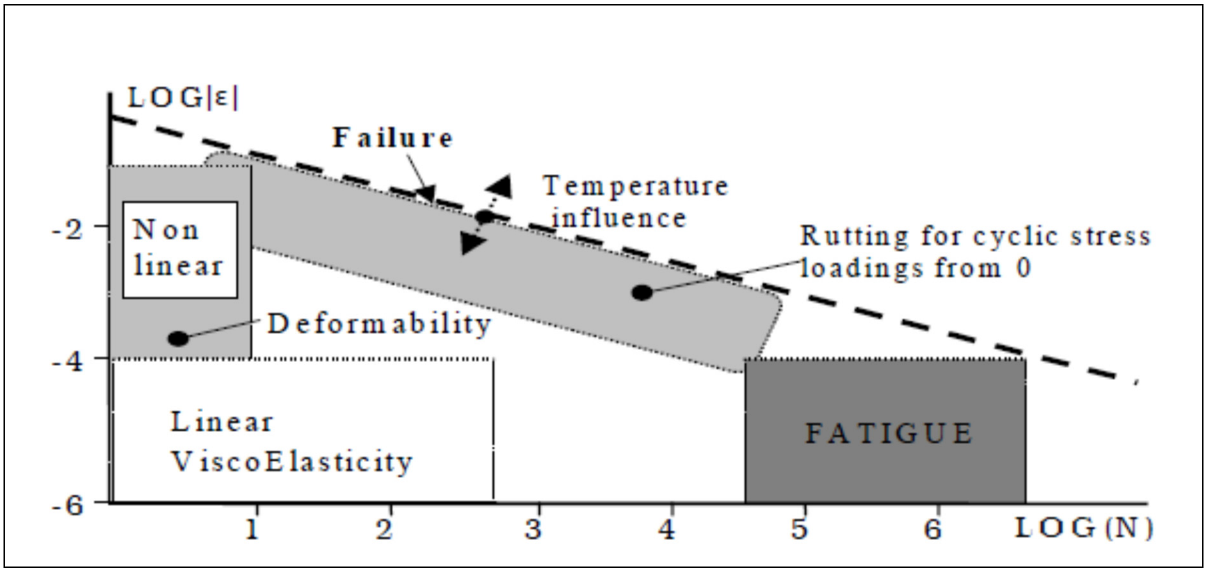


Figure 1.9. Typical cold mixture behaviour as a function of applied cyclic force and strain Taken from Di Benedetto, Partl, Francken, and Saint André(2001).

Since cold materials are a mix of bituminous material and aggregate, usually *complex modulus* E* is used for evaluation of linear viscoelastic behaviour of the asphalt mixtures at various temperatures. By definition complex modulus is the proportionality coefficient between sinusoidal complex amplitude of the stress, for a given frequency and amplitude of the strain (Carter & Perraton, 2002). It can be measured through tension-compression test (illustrated below). The oscillatory loading to the asphalt can be considered in several ways as depicted in Figure 1.10. Typically, the methods should be selected based on the assessed parameters and desired properties. Shear testing and axial testing are the two main groups of tests performed in this field of study which are usually conducted in strain control or in stress control, respectively. Several tests, like tension strength, compression strength of simple shear loading (SST) dynamic shear rheometer (DSR) is some of the performed tests to measure directly or indirectly different mechanical properties of the final mixture. However, the lack of standardized protocol for different temperatures and climatic situation is still needed in the literature. For instance, Lesueur and Potti (Lesueur & Potti, 2004) mentioned that future investigations for test procedures concerning the prediction of the in-situ density of cold mixes and fatigue behaviour, workability and accelerated curing methods and measurements in the laboratory studies should be developed.

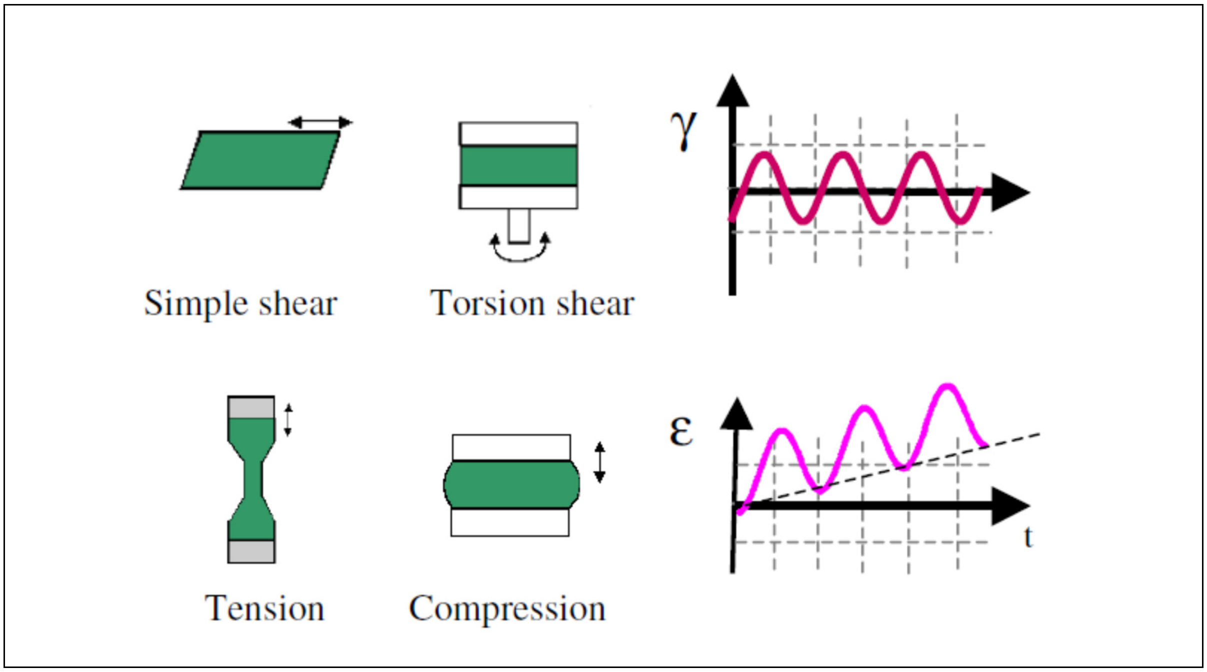


Figure 1.10 Methods of applying cyclic loads
Taken from Godenzoni (2017).

1.7 Additives

In this section, the general criteria for the use of additives in cold recycling mixtures with bitumen emulsion are presented. Different properties of recycled mixtures with bitumen emulsion such as physical, durability and mechanical characteristics, concentrating on the influence of additives to the mixture and binder are presented. Generally, there are two types of additives including:

- Additives incorporated in the mixture,
- Additives incorporated in the binder.

Lime, cement, waste pozzolans, and chip seals are the most well-known additives in the first group, illustrated as follows:

1.7.1 Lime`

- **Lime and hydrated lime** are extensively used to increase the mixture durability and accelerate curing process, respectively (Godenzoni, 2017). According to the Zeta measurements lime affecting the droplet interaction renders the cationic charge in the emulsion. 1-3 % of RAP weight is the normal range of lime content in the recycled materials (Kim & Lee, 2012). According to the literature usage of lime increase the mixture density while it can severely reduce the moisture sensitivity of cold recycled materials with bitumen emulsion (increasing the moisture resistance) (Ayar, 2018). Moreover, lime or its slurry can effectively increase the mechanical properties of mixture such as tensile strength, dynamic modulus, marshal stability, rutting resistance and resistance to the permanent deformation and stiffness (Niazi & Jalili, 2009). It should also mention that the lime usage can prevent the pollution problem at the project site.
- **Cement** traditionally, 1-3% cement by weight of the RAP is used in cold recycled materials with bitumen emulsion (also called cement-treated materials). The reaction between cement and bitumen emulsion controls the main effect of cement on the mixture performance. Addition of cement can increase the stiffness of recycled materials and also accelerate the emulsion breaking. It results in rough texture and a pitted surface and better temperature stability. It can also increase the mechanical properties of the recycled materials (such as unconfined compressive strength, rutting resistance and resilient modulus); however, reduction in density and compatibility as a result of cement addition should be expected. The RMBEs were manufactured by three types of bitumen emulsion including Cationic Slow Setting (CSS-1), Cationic Medium Setting (CMS-1) and High Float Anionic Medium Setting (HFE-150). Due to the fact that the cement-emulsion increases the cohesiveness of the mix higher moisture resistance is reported as a result of cement addition (more than 0.5 %) (Niazi & Jalili, 2009). Generally, mechanical properties of the cold recycled materials with bitumen emulsion increase by increasing the curing time and cement content as shown in Figure 1.11 (Kavussi & Modarres, 2010). It should be noted that when the cement content surpasses a specific limit, more brittle behaviour

under low temperature should be expected (Kavussi & Modarres, 2010). Besides, fatigue behaviour of the mixture should be cautiously regarded.

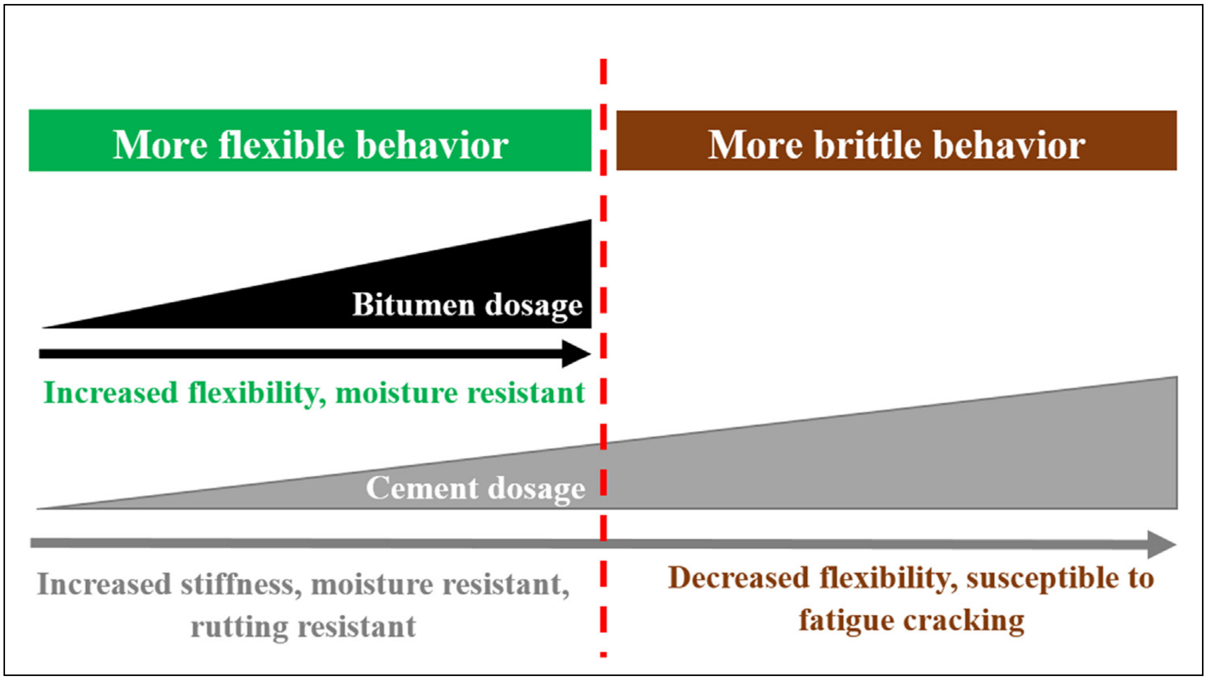


Figure 1.11 Effect of cement usage in cold recycled materials in a summarized view Taken from Kavussi and Modarres (2010).

- **Waste pozzolans**: Different waste materials such as coal waste. Coal waste ash, fly ash and furnace slag can be effectively used in cold recycled materials. As it is demonstrated by their name, adding this material will add the pozzolanic nature of the mixture. However, influence of their addition to the mixture on the mechanical performance of the materials is not clear and several studies showed different behaviour for different types of waste materials.
- **Chip seals** are mixes consisting of asphalt emulsion and aggregate that are usually added to the existing roadways to slow the deterioration process and improve the surface quality of the pavement. According to the literature, the properties of the asphalt emulsion had a direct impact on the performance of cheap seals (Castorena, Ilias, Adams, & Kim, 2018). Therefore, emulsion performance-graded (EPG) is used to evaluate chip seals. This

parameter evaluates the aggregate loss and bleeding, which occur at intermediate (or low) temperatures and high temperatures, respectively. Castorena et al. (Castorena et al., 2018) in their experimental study investigate the EPG for low temperatures. They suggested crossover modulus as the most promising evaluation of emulsion (or emulsion performance grade, EPG) at low temperatures.

On the other hand, polymers and rejuvenators are the most well-known additives incorporated in the binder.

- **Polymers** are used in some highway agencies to provide a polymer modified bitumen emulsion. Different polymers were used in different countries to increase the performance of bitumen emulsion in cold recycling materials (Ayar, 2018). Different researchers used different polymers like SBS, CSS1, HFMS-2s and HFMS-2p. Results indicated that polymer treated bitumen emulsion in general had lower void content, better rutting resistance and better marshal stability. However, the polymer could not demonstrate a positive effect on the tensile strength and moisture sensitivity. It also increases the mixture viscosity. Some mix treating (cement-polymer) projects shoed better mechanical performance (Ayar, 2018).

1.7.2 Rejuvenators

Asphalt pavements lose their mechanical properties and needs treating as they age. As mentioned above the main aging mechanism of asphalt binders is oxidation and loss of volatiles resulting in having a stiffer binder in the asphalt (Holleran, Wieringa, & Tailby, 2006; Zhang, Wang, You, Jiang, & Yang, 2017). The interest on the usage of reclaimed asphalt pavement (RAP) has been growing significantly. Specifically, RAP employment in the cold recycling of asphalt has been considered as a good alternative for the hot recycling for the sake energy saving and emission reduction. However, this method due to high amounts of bitumen emulsion and technical issues is expensive for the small site recycling. Besides, aged RAP bitumen has undesirable high stiffness and low creep rate which makes it venerable to low temperature thermal cracking (Copeland, 2011; Yu, Zaumanis, Dos Santos, & Poulikakos, 2014). In other words, high amount of RAP in the mixture (which is desirable from energy conserving and cost point of view) results in very stiff mixes which are difficult to compact and susceptible to unexpected permanent failure (Copeland, 2011). Several techniques have been evaluated to facilitate the usage of RAP in the asphalt mixes in the literature. That includes mixing with softer virgin bitumen, higher asphalt content mixture, warm mixing technology; however, none of those are suitable for high amount of RAP content in the mixture (Im, Karki, & Zhou, 2016). Rejuvenators can reactivate the RAP binder and bitumen in the mix so that a lower amount of bitumen during the process of cold recycling of asphalt materials to be needed. Rejuvenators can restore the characteristics of the aged bitumen partially or fully by reactivating the binder (restoring the aged properties to its original state). Rejuvenation is a transient (time dependent) and temperature dependent phenomenon. However, finding economic and environmentally friendly rejuvenators are difficult and need comprehensive study and experimentation. There are several used rejuvenators in the literature which have been used to reduce the asphalt binder stiffness and provide better road performance (Nahar et al., 2014; N. Tran et al., 2017; Hainian Wang, Zhang, Chen, You, & Fang, 2016; Xie, Tran, Julian, Taylor, & Blackburn, 2017; Yu et al., 2014; Zaumanis, Mallick, Poulikakos, & Frank, 2014).

1.7.2.1 Rejuvenators' Content

The logic behind the usage of rejuvenators comes back to the composition of asphalt. As mentioned asphalt is composed of four different chemical fractions: asphaltenes, resins, aromatic and situates (Elkashef, Podolsky, Williams, & Cochran, 2017). The combination of the first three items are referred as maltenes. The asphaltenes, with high molecular weight, forms a colloidal suspension in the low molecular part of the binder or maltenes (Elkashef et al., 2017). Firoozifar et al. (Firoozifar, Foroutan, & Foroutan, 2011) reported that the an increasing in the asphaltene content (which was achieved by adding extra propane asphaltene tar (PDA)) led to significant increase in the penetration index which is of great interest as it has a similar influence of the aging. Resins were also reported to have an important effect on the controlling the dispersion of asphaltenes and asphalt viscosity (Bitumen, 1995). On the Other hands, addition of maltenes helps to rebalance the aged asphalt chemical composition which lost a lot of maltene as a result of oxidation (during the aging maltenes fraction of asphalt is converted to the viscous asphaltenes) Therefore, rejuvenators containing high portion of maltenes are good choices to this end. Rejuvenators which are usually added to the mixture diffusing within the aged bitumen contribute to the softening of the asphalt mix with RAP content. This diffusion usually starts with a coating of RAP content and continues by gradual seep into the aged bitumen layer an finally diffusing through the film thickness (Carpenter & Wolosick, 1980). It should be mentioned that the rejuvenation does not mean the reversing the oxidation process of aged bitumen, rather it means the restoration of viscoelastic properties of the binder.

1.7.2.2 Rejuvenator Dosage and Source

It should be mentioned that the overall performance of the rejuvenators is source dependent and even the commercial rejuvenators need to have comprehensive investigation about their optimum content in the mix when it comes to the different RAP and mixing procedures according to the pre-defriend standards in each region (Zhang et al., 2018). This is also in correlation with the chemical composition of different bitumen. While, a specific rejuvenator could result in positive improvement on the mechanical properties of an asphalt with a specific bitumen, it could have not desirable influence on the other. However, researches through different sources, size may give insight to have general proceedings to find out solution in each case. For instance, Yu et al. (REF) reported that the effect of rejuvenator was more prominent on the ABD bitumen (PG 58-28) compared to the ADD bitumen (PG 58-10). The authors suggested that the reason for such observation could be elaborated on the better chemical reaction of rejuvenator with ABD bitumen. In addition, the durability of the rejuvenation is an important parameter in the selection of the proper rejuvenators in the mix. Upon volatilization of softening agents, which usually contain volatile components, no additional enhancement is available for the mixture. Recently, Mohamdafzali et al. (Mohammadafzali et al., 2015) in their study on the long-term performance of different rejuvenators, showed that their performance significantly differs with respect to their durability.

The selection of rejuvenator dosage important influence on the mechanical properties of asphalt was mentioned in several studies like Shen et al. (Shen, Amirkhanian, & Tang, 2007) who suggested that usage of blending charts to obtain an optimum dosage of rejuvenator content in the mix. Optimum dosage determination is of crucial importance because higher content would result in unwanted excessive softening which in turn contribute to the mechanical performance problem such as rutting, stripping and mix instabilities. Zhang et al. (Zhang et al., 2018) in their experimental study on the usage of rejuvenators derived from waste wood showed that the rutting resistance and fatigue resistance of the aged asphalt binder were restored using the selected rejuvenator. The optimum concentration of 15% was recommended for the best performance on the rheological properties of the asphalt (Zhang et al., 2018).

1.7.2.3 Bio Based Rejuvenators

Several rejuvenators such as petroleum based aromatic extracts, distilled tail oil, and organic oil have been suggested in the literature (Elkashef & Williams, 2017). Among the variety of choices for the origin of bio-based rejuvenators have been attracted a significant interest due to their availability and environmentally friendly nature (Lei, Bahia, & Yi-qiu, 2015; Yang, You, & Dai, 2013; Zhang, Wang, Gao, You, & Yang, 2017). Bio oils are generally produced by fast pyrolysis technologies resulting in three main components including bio-chars, gasses and liquid. In this process biomass materials in the absence of air are heated rapidly to 450-600 °C then a rapid cooling are designed for the vapour in less than two seconds (Portugal, Lucena, Lucena, & Beserra da Costa, 2018). While the chars and gasses can be reused (as an energy source). The last item (liquid) is mainly considered as bio-based rejuvenators. Previous researchers proved the positive impact of the bio-based rejuvenators on the improvement of low asphalt binder low-temperature performance (HG Wang & Junfeng, 2014; Yang & You, 2015; Yang et al., 2013). Most of the studies focus on the influence of rejuvenators addition on the stiffness of the aged bitumen and low temperature cracking resistance of the asphalt (Elseifi, Mohammad, & COOPER III, 2011; Hajj, Souliman, Alavi, & Salazar, 2013). The general conclusion of these studies states that the rejuvenators positively reduce the aged bitumen stiffness and significantly improves the low temperature cracking resistance (Mogawer, Booshehrian, Vahidi, & Austerman, 2013; N. H. Tran, Taylor, & Willis, 2012). Various kind of bio-based rejuvenators like waste vegetable oil, organic oil and distilled tall oil can be used in the cold asphalt recycling method. For example, the bio-oil rejuvenator driven form biodiesel residue was reported to increase the low temperature crack resistance and workability of the aged asphalt. According to Gong et al. (Gong, Yang, Zhang, Zhu, & Tong, 2016) this happened due to the fact that the oil compensate the loss of light components in the mixture. That is why, bitumen emulsions treated with rejuvenators are generally containing different oils like maltene oil or vegetable-based oil (Elkashef et al., 2017).

Soybean oil-based rejuvenators were also considered as effective rejuvenators in serval studies. The abundance of soybeans in the US is the main reason for the research in this field. The United States produces one third of the world soybean (Elkashef et al., 2017) which is mainly used for soybean meal or oil production. Seidel et al. (Seidel & Haddock, 2014) studied the effect of different dosages of soybean acidulated soap stock (SAS) on the mechanical performance of the asphalt mix. A consistent decrease on the critical high temperature was reported with respect to the SAS modification while and enhancement in the low temperature was also observed. Successful experiences of soybean oil was also reported in the warm-mix asphalt mixtures (Portugal et al., 2018).

Considering the organic nature of vegetable oil-based rejuvenators and bitumen inside the asphalt (in other words their compatibility), vegetable rejuvenators (as a bio-binder) can be a good alternative for the fuel-based rejuvenators. Usage of vegetable-based oils, not only from the environmental point of view, but also concerning the process budget attracted the researcher's attention recently. For instance, Hugener et al. (Hugener, Partl, & Morant, 2014) in their extensive (laboratory and in situ) paper evaluated the usage of vegetable-based oil rejuvenators in bitumen emulsion of cold recycled materials (See Figure 1.12). In the paper, a new cold recycling process for small on-site pavements using vegetable oil-based rejuvenators was presented. Three main types of rejuvenators including, rapeseed oil, linseed and used cooking oil and their combinations were evaluated and compared with commercial rejuvenators. Several parameters including the amount of rejuvenators, curing time and temperature, water concentration, time between mixing and compaction, compaction method, cement concentration and mixing procedures were studied. Results were analyzed based on uniaxial compression test (UCT) to evaluate the UCT resistance as the main target mechanical property of the recycled asphalt (Hugener et al., 2014). Comparing the results of mobile load simulator (MMLS3) in the in-site experiment with the fast UCT test in the laboratory revealed the reliability of UCT test for data analysis. In the case of specimen preparation methods, results indicate that gyratory compactors are better than Marchal hammer for compaction.

According to the authors 14 days of curing time (water evaporation time) at 40˚C is the optimal accelerated curing process. Besides, addition of cement or hydrated lime could accelerate the drying time, but jeopardize the ultimate mechanical properties of the asphalt. It was found that the mixing procedure does not have significant effect on the final UCT-resistance. Nevertheless, considering the hydrophobic nature of rejuvenators it is recommended to add rejuvenators to the mix before the water to have slightly better ultimate UCT (Hugener et al., 2014). An observable trend showing the small reduction of mechanical properties of the asphalt with delay on the compaction was found for commercial rejuvenator in the laboratory. Nonetheless, the opposite trend was found for the rapeseed oil in the field test, which showed the importance of more studies on the reaction of rejuvenators with the asphalt binder.

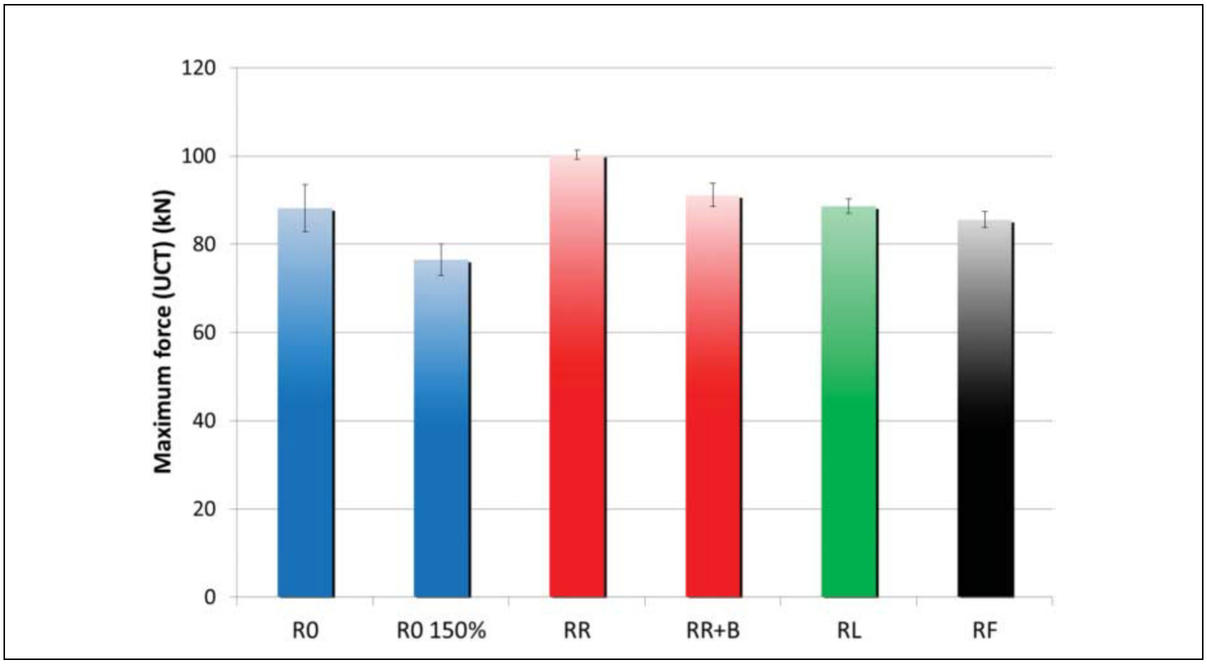


Figure 1.12. UCT-resistance for different mixes (60 days curing). (R0: commercial rejuvenator, RR: rapeseed oil, RL: linseed oil, RF: frying oil, +B: plus bitumen.

Taken from Hugener et al.(2014)

Both laboratory and in-site experiments confirm the performance of cold recycled asphalt with vegetable oil rejuvenators. However, the cold recycled asphalt has lower performance in comparison to the hot mix asphalt against water content. Results indicated that drying the asphalt would restore its performance. Therefore, the authors recommend a good drainage for the cold asphalt to increase its durability and performance (Hugener et al., 2014). Results also show that vegetable oil-based rejuvenators are considerable options for reactivation of the old binder in the reclaimed asphalt (in the cold mixing recycling method). Employment of used cooking oil rejuvenators as well as the other vegetable oil rejuvenators resulted in the nearly same UCT-resistance of commercial rejuvenator. Rapeseed oil providing 10% higher UCT-resistance showed maximum performance. However, it was found that other types of rejuvenators, including waste cooking oil (gathered from restaurants), had nearly the same performance. Assessment of rejuvenator amount of the mixture showed that content higher than 50% could reduce the UCT significantly due to viscosity reduction. Therefore, considering the conclusion that the amount of vegetable oil rejuvenators is more important than their type, waste cooking oil rejuvenator identified as an economic and green option for cold asphalt recycling (Hugener et al., 2014). Waste cooking oil, according to Zargar et al. (Zargar, Ahmadinia, Asli, & Karim, 2012), was also reported to increase the rheological performance of the asphalt with a penetration grade of 40/50 to those of asphalts with a penetration grade of 80/100. Zaumamis et al. (Zaumanis et al., 2014) also showed that that the vegetable oil rejuvenator with a concentration of 12 wt% could significantly increase the cracking resistance performance of the asphalt.

1.8 Mixture preparation

All the cold recycled materials should proceed through a preparation process to be used in the road pavements. The section starts with the introduction of a rational mix design procedure and continues with the introduction of the most important procedures including, curing, compaction and mixing steps and their relative impact on the final product performance.

To start the mixing design, quality and quantity of a mix components (including, aggregate, bitumen emulsion and water) should be tightly controlled. Then the process should follow the design steps including defining of:

- Aggregate gradation
- Minimum water content
- Emulsifier type and desired mix PH
- Emulsifier content
- Minimum emulsion content
- Desired mechanical properties

To achieve a final mix the specimen should be prepared and evaluated after the first part of the flowchart definition (before mechanical properties evaluation). Producing a cold mix usually take place through a sequence of actions including mixing, compacting and curing which illustrated in next sections.

1.8.1 Mixing sequence

According to the literature, sequence of adding different components of cold mix materials can play an important role in the final acquired mechanical properties. Generally, the two main components of recycled cold mix materials are RAP and water. However, in different cases other additives like rejuvenators can be added in different stages to the mix. (See figure 1.13).

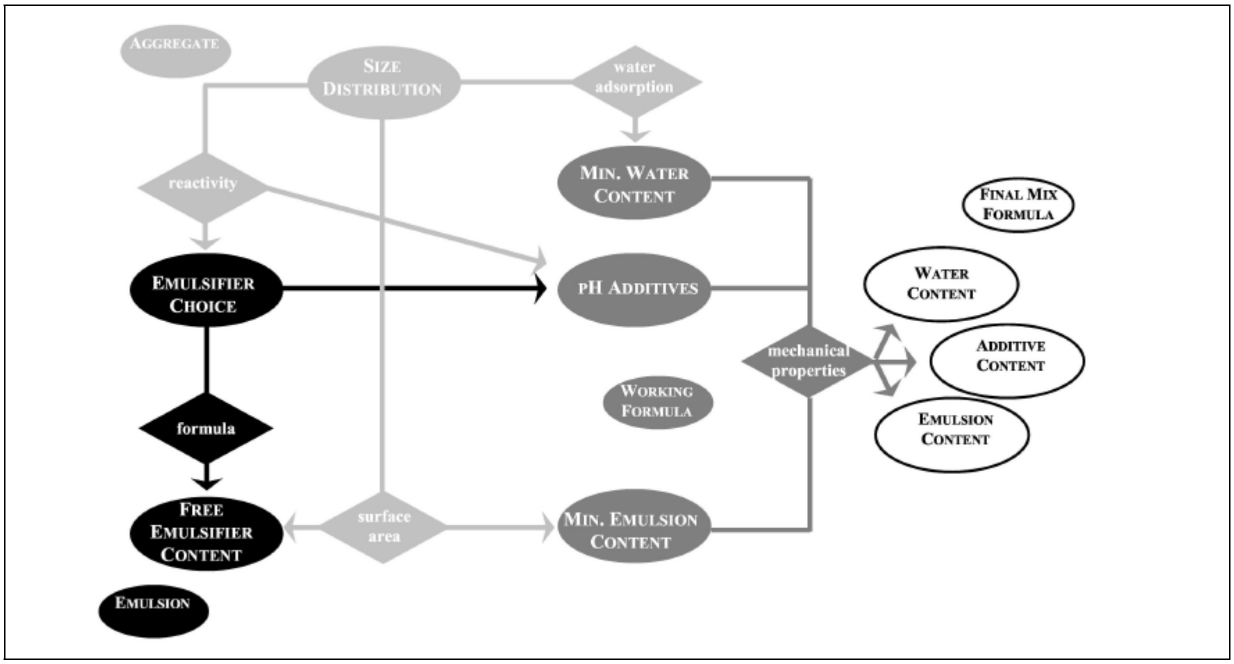


Figure 1.13. Mix design flowchart
Taken from Godenzoni (2017).

Hugener et al. (Hugener et al., 2014) in their work using rejuvenators came up with the idea that the mixing steps could not change significantly the final mechanical properties of the mix.

However, according to their results adding the rejuvenators before the water appeared to give better results (Figure 1.14). They illustrated this phenomenon using the hydrophobic nature of rejuvenators. They mentioned that the more water added to the wet RAP makes the diffusion of rejuvenators to the RAP binder difficult. Therefore, they recommended that the rejuvenators should be added to the dry RAP. In the case of compaction time, they mentioned that although the compaction time should be cautiously regarded, the delay in compaction could not change significantly the final product properties. However, they mentioned that in the field test they observed that the mechanical properties of the asphalt decreased after a long time of storage before compaction. They recommended more investigation in this case study.

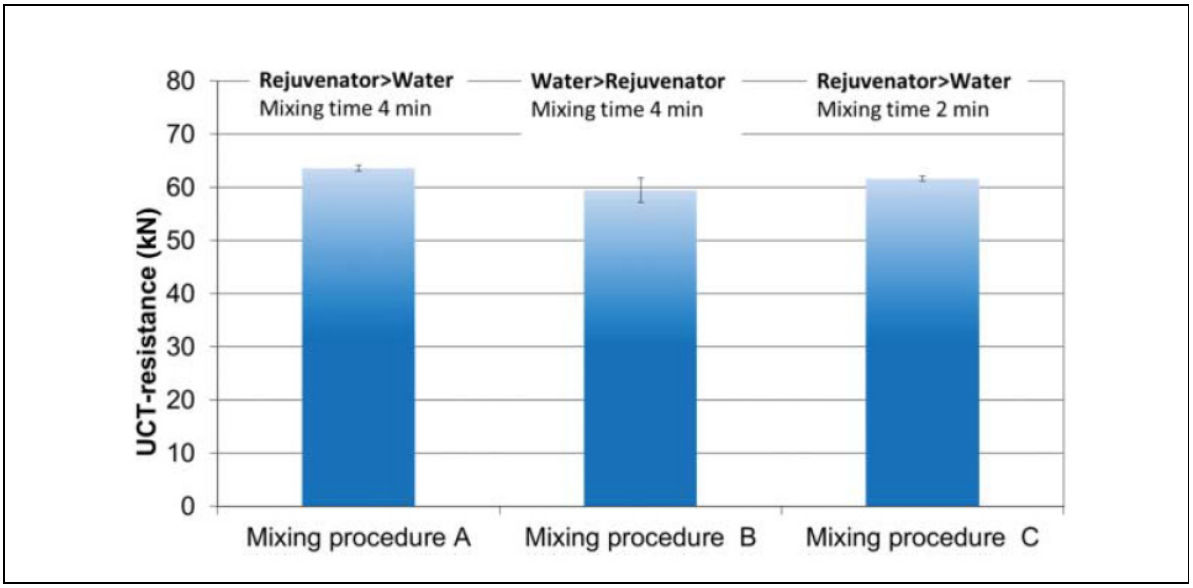


Figure 1.14. Comparison between sequence of water and rejuvenators addition to the mix Taken from Hugener et al. (2014).

1.8.2 Compaction

Compaction is a fundamental step for improving particle contacts and reducing voids in the cold material mixing production process. It directly affects the achieved density of the mixture which in turn is critical for the final performance of the mixture (Godenzoni, 2017).

There are several methods of compaction in the literature. Several laboratory compaction techniques are used to emulate the particle orientation after rolling. In other words, compaction of asphalt material can help to position the particles of the mix and redistribute the binder to make a continuous film. However, too much compaction can cause damage to the asphalt mixture (Gandi, 2018). It should be noted that the expected density of the field is usually not satisfied and further compaction should be performed to have the desirable density of the field. Table 1.2 provides an overview of the several compaction methods that can be performed aside from the standard Marshall compaction. According to the literature an ideal compaction method is not identified yet, however, mentioned methods with their relative parameters showed promising results. It should be noted that the compaction method should be selected based on both the volumetric and engineering properties of the target mixture to recreate the desired field density as close as possible.

Several investigations made valuable comments about compaction in the literature. Hugener et al. (Hugener et al., 2014) stating the study of Holl (Holl, 1991) mentioned that the Marshal compaction is not good for the specimen with high amount of water (like the case of cold recycling with bitumen emulsion using rejuvenators). This is due to the fact that in an oversaturated specimen where the water volume is higher than the air content in the structure, the water acts as an elastic spring during the impaction by Marshall hammer (not enough drainage). On the other hand, gyratory compaction as a static method water has enough time to flow out of the mould. Hugener et al. (Hugener et al., 2014) using an experimental procedure also investigated two possible compaction methods in their case study (see Figure 1.15). According to the authors, even with employing of 100 impacts per side in Marshal hammer technique they came up with the 15 % of air void which is higher than desired 12 % content. On the other hand, target 12 % of air voids could be achieved using 55 gyrations and water flowed out from the mould through constant kneading action. The same approach was reported by Kim et al. (Kim & Lee, 2012) who used 25 gyrations for their extensive study of curing process.

Table 1.2. Summary of laboratory compaction techniques and their relative employed parameters in several studies
Taken from Gandi (2018).

Compaction method	Settings/Temperature	Binder
Kneading compactor	Ambient temperature	Foamed bitumen
Gyratory compactor	Angle = 1° Pressure = 1.38 MPa	Foamed bitumen Bitumen emulsion
Texas gyratory compactor	25 °C	Foamed bitumen
Gyratory	20 revolutions Pressure = 1.38 MPa	Foamed bitumen
Gyratory compactor	150 revs, Angle = 2°, Pressure = 0.24 MPa (100 mm diameter) 150 revs, Angle = 3°, Pressure = 0.54 MPa (150 mm diameter)	Foamed bitumen Bitumen emulsion
PCG (French gyratory compactor)	200 cycles with LCPC carousel	Bitumen emulsion
Vibratory hammer compaction.	Power rating 1500 W Freq. 15 – 31.5Hz Point Energy 25 J	Foamed bitumen Bitumen emulsion
Shear gyratory compactor	EN 12697 -31	Bitumen emulsion
Static compaction	Compaction force of 25 kN for 3 mins	Bitumen emulsion

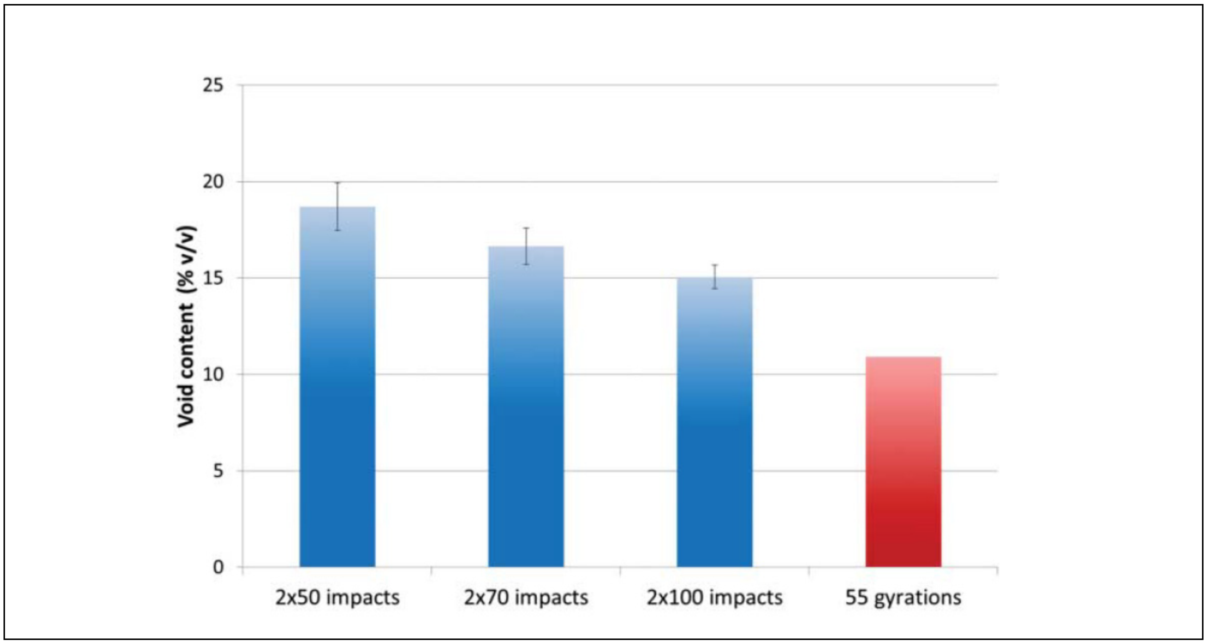


Figure 1.15. Comparison between Marshal and gyratory compaction
Taken from Hugener et al. (2014).

Gandi (Gandi, 2018) in his thesis mentioned that the compaction is easier at lower temperatures (like 5 or 10 degree C) in comparison to the regular 23º C. They concluded that the RAP has more a black rock behaviour and form somewhat rounded shape at lower temperatures (like Quebec region) which makes the compaction easier.

Ayar (Ayar, 2018) in his study investigating the additives to the cold recycled materials mentioned that the compatibility of additives is a key parameter for their selections in the mixture. Serfass et al. (Serfass, Poirier, Henrat, & Carbonneau, 2004)mentioned that increasing the gyratory compaction degree will result in indirect tensile modulus growth. Besides, increasing the compaction gave higher unconfined compressive strength in their work.

It should be noted that for the laboratory tests the specimens are very sensitive to handle right after the compaction (Hugener et al., 2014), when the cohesive strength is not developed yet. Two hours after compaction, the stability of the gyratory specimens is reported being sufficient to unmold the specimens. Still, the wet specimens are very fragile and had to be handled with extreme care, especially for being transported to another place (See figure 1.16).

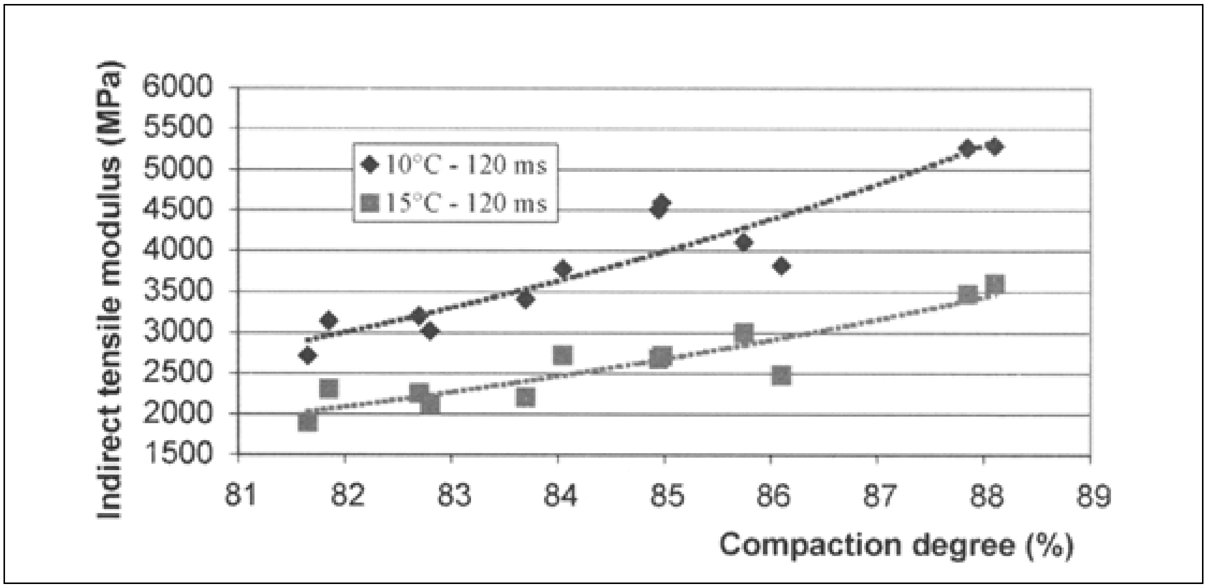


Figure 1.16. Influence of compaction stiffness
Taken from Hugener et al. (2014).

1.8.3 Curing

Curing is a process in which moisture content of asphalt mixture should evaporate from the binder structure to achieve the desired stiffness (as the moisture content in the mixture decreases with time the strength and stiffness increase) (Gandi, 2018). Laboratory and field tests showed that the strength and stiffness of a cold mixture are relatively low after initial preparation (compaction and mixing). Generally curing is the dependent variable of some independent variables like humidity, temperature, compaction level, etc. As it can be seen these variables are the environmental situation in which the curing process take place. Typically, curing continues up to 6 – 18 months in the real-life road pavement recycling with cold materials. This long time of curing makes the laboratory investigations difficult to carry out. Currently, different agencies employ differing moisture content. One of the usual industrial standards is either a maximum moisture content of 1.5% or a curing time of 10–14 days (Kim & Lee, 2012). However, these standards should be modified according to the environmental situation (rain, snow, temperature) in each region. Therefore, several studies were devoted to developing a standard laboratory mechanism of cold mixture curing process and field tests in this context. Several studies investigated the curing time and temperature effect on the different parameters of cold recycled materials. Serfass et al. (Serfass et al., 2004) mentioning the evaluative nature of the cold materials in their early life investigated the influence of curing on cold mix mechanical performance and a proposed a curing procedure to the industry. According to them, the cold mix materials should be evaluated in at least three stages of fresh, mature and aged asphalt. Emulsion asphalt during the curing time can alter in aggregate-emulsion reactivity; binder film coalescence and cohesion build up. Their results demonstrated that increasing the curing time and temperature reduces the moisture content and increases the unconfined compressive strength. After evaluation of different times, temperature and humidity they proposed 14 days of curing at 35 ºC and 20% of humidity. According to the author this process is equivalent to the 1 to 3 years of filed curing under temperature climate (Serfass et al., 2004). Hugener et al. (Hugener et al., 2014) in their extensive study of rejuvenator effects on the cold mixture performance stated that a curing temperature of 40ºC during a period of 14 days can be considered as an optimal process for laboratory testing acceleration. According to the author similar to cold asphalt with bitumen emulsions, a certain time period (curing time) is required for the cold recycled asphalt treated with rejuvenators to evaporate the water content in the mixture and for the rejuvenator to diffuse into the old bitumen. In other words, for the bituminous emulsion the curing time is used not only for the conventional water evaporation, but also for providing a time for the rejuvenators to act effectively. It is important that the curing procedure does not cause any deterioration in the specimen and reflects real-field behaviour as closely as possible. The curing progress was studied both by the change of mixture weight due to water loss (water evaporation) and by the UCT-resistance experiments. According to the author, most of the curing process occurs in the first three days after the compaction, while the rest take place with similarly same speed. They reported a linear relation between water loss and UTC improvement (See figure 1.17).

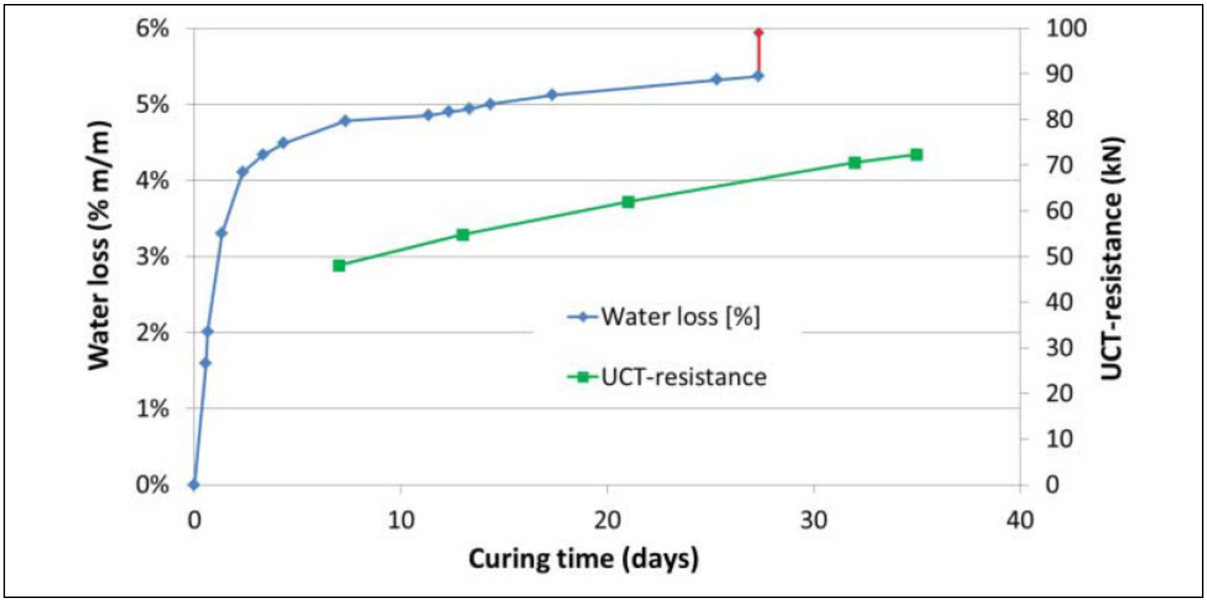


Figure 1.17 Curing process and UTC improvement analysis
Taken from Hugener et al. (2014).

The authors assumed that the evaporation of water will continue with the same speed for the rest remaining water (20 days of curing time was calculated). However, they had some contrary results showing the complexity of the curing process and its several parameters that need to be considered for a comprehensive analysis. Author mentioned that as the temperature increases the process is faster. The process for 40ºC is about three times faster than regular 23ºC. However, this correlation between the temperature and the time of curing is not linear and increasing the temperature to 50ºC could only increase the speed marginally (See figure 1.18).

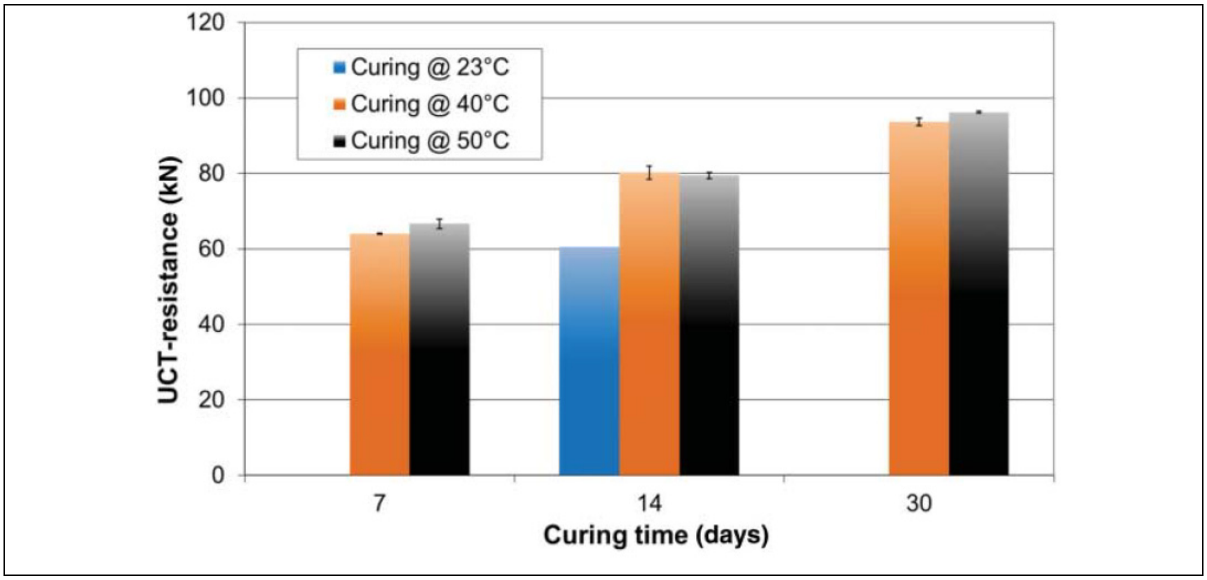


Figure 1.18. Effect of curing time and temperature on the UCT resistance of cold materials Taken from Hugener et al.(2014).

Cardone et al. (Cardone, Virgili, & Graziani, 2018)analyzing the bond strength between bituminous binder in reclaimed asphalt aggregate and bitumen emulsion composites made several comments on the effect of curing. According to the authors, curing conditions could not affect the cohesive interaction between residual bitumen and aged bituminous film coating (the bond strength). They mentioned that the curing temperature and time effect are mainly visible on pull-off tensile strength (POST) of a system with mastics (up to 10%) rather than emulsion (Cardone et al., 2018). Pooyan Ayar in the paper discussing the effect of additives on the mechanical performance of recycled mixtures stated that during the early stage of curing, the interface between bitumen emulsion and RAP aggregates was a susceptible point for fractures due to the existence of a water membrane (Ayar, 2018). The same observation of increasing of the UCT was observed for the resilient modulus as the curing time was increasing.

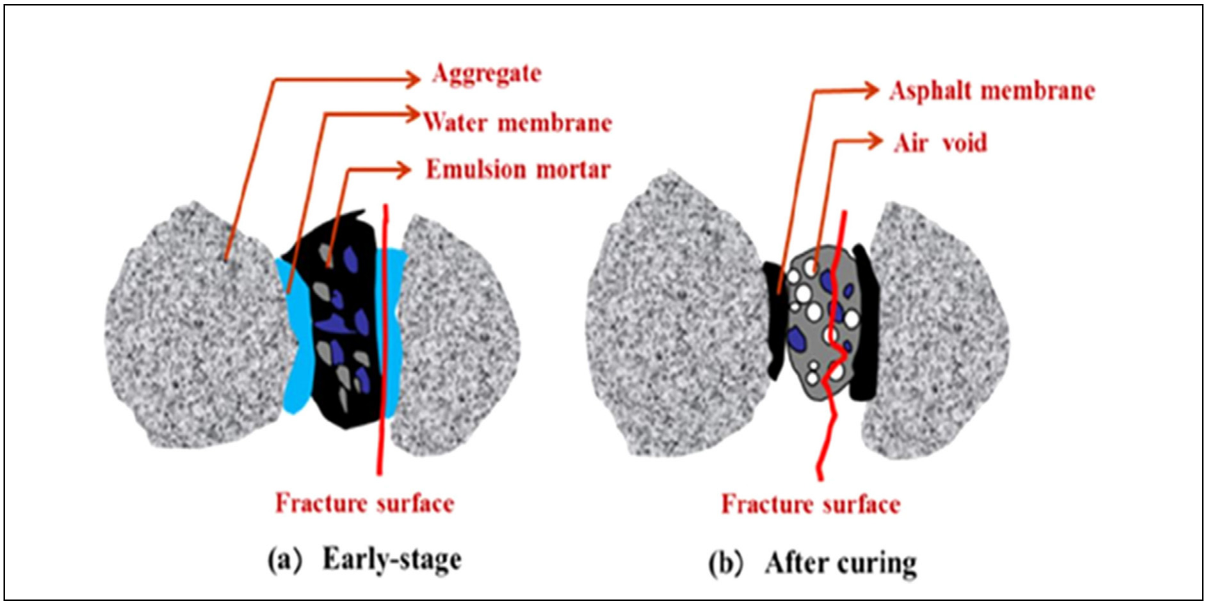


Figure 1.19. Mechanism of strength growth in recycled materials having additives in the binder

Taken from (Ayar, 2018).

In another reputable study, Kim et al. (Kim & Lee, 2012) in their research investigated the curing time and temperature effect on the mechanical properties of CIR (indirect tensile strength, dynamic modulus, and flow number) using foamed and emulsified asphalt. According to the author, amount of moisture content in the mixture and curing time can severely affect the mechanical properties of the CIR materials. However, the effect for the foamed asphalt was more than the one for the emulsified one. Submerging a cured CIR in water for 24 hours (similar to the environmental condition of the region with high amount of annual rain or asphalt who has experienced rain during the transportation) could significantly reduce the indirect tensile strength of the material (Kim & Lee, 2012).

To finish the mixing procedure section an overall schematic view of the resistance (Re) evolution during the time after compaction and curing is presented in Figure 2.18. As it can be seen and reported by the researchers in the literature, physical state of the cold materials evolves with time according to the moisture (water) content (w%) because of both evaporation and hydration process. Therefore, in any research in this field of study a rheological study from the early stage of the cold mix to the long-term (final) one should be carried out. Generally, the long-term state also called as steady-state or curried state in the nomenclature. The long-term behaviour of the material is the desired/defined final mechanical properties of the cold mix that should be taken into account by the engineers and road paving agencies.

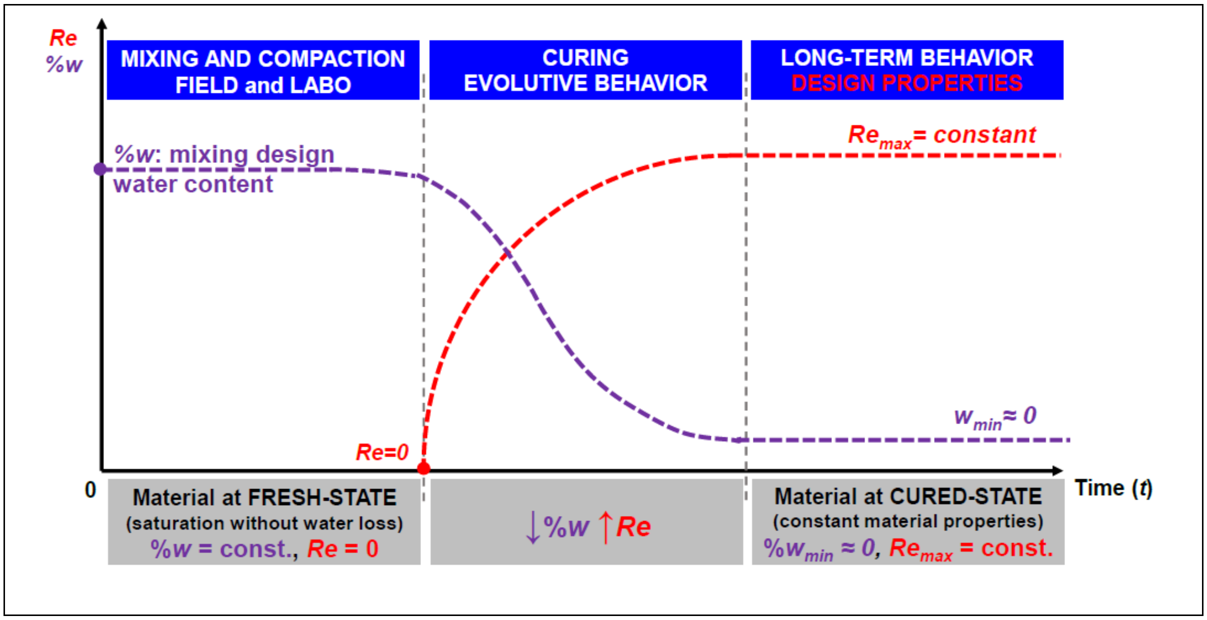


Figure 1.20. Transient evaluative behaviour of cold materials mixture
Taken from Ayar (2018).

1.9 DSR Concept and Background

Asphalt binders are viscoelastic meaning that they behave partly like an elastic solid and partly like a viscous liquid. In the elastic solid-state deformation due to loading is recoverable or in other word asphalt binder is able to return to its original shape when a load is removed, while in the viscous state such a deformation is non-recoverable, and the binder cannot restore its original shape after removing the load. DSR which has been used in the plastics industry for years, is capable of quantifying and determining both elastic and viscous properties of the materials. Such capability makes DSR a well-suited method for characterizing asphalt binders in the in-service pavement temperature range used in the pavement technology.

The DSR test works based on the measurements of a specimen's complex shear modulus (G*) and phase angle (δ). Where, the complex shear modulus (G*) can be taken into account as the specimen's total resistance to deformation as a result of repeatedly sheared tensions, while the phase angle (δ), corresponds to the lag between the applied shear stress and the resulting shear strain (Figure 2.13). In such calculation, the larger the phase angle (δ) indicates the more viscous the material. It should be mentioned that the phase angle (δ) limiting values are:

- $\delta = 0$ degrees, corresponding to purely elastic material
- $\delta = 90$ degrees, corresponding to purely viscous material:

In this test a specified DSR oscillation rate of 10 radians/second (1.59 Hz) was used for the experiments. This value represents the physical simulation of the shearing action corresponding to a traffic speed of about 55 mph (90 km/hr).

1.9.1 DSR Superpave Specification Logic

G* and δ as the key parameters in the DSR test are used as predictors of HMA rutting and fatigue cracking. Therefore, both short term and long-term resistance of the asphalt are considered in this test. It should be mentioned that the rutting is the main concern early in the pavement life, while fatigue cracking becomes the major concern in the long-term life.

1.9.2 Rutting Prevention

Having a good rutting resistance means that an asphalt binder should be stiff (so that it does not deform too much) and it should be elastic (to be able to restore and return to its original state and shape after load deformation). Such a property requires a large complex shear modulus elastic portion, G*/sinδ (Figure 1.14). Therefore, during the HMA pavement's early and mid-term life when the rutting is of significant concern a minimum value for the elastic component of the complex shear modulus is specified. Intuitively, the higher the G* value, the stiffer the asphalt binder is (or, in other words, it is able to resist deformation), and the lower the δ value, the greater the elastic portion of G* is (i.e it is able to recover its original shape after being deformed by a load).

In another point of view to this phenomenon, it should be considered that rutting is basically a cyclic loading phenomenon. This means that in each traffic cycle, a work (stress) is being done (applied) to deform the pavement surface. The elastic rebound of the pavement surface recovers part of this work, while the remaining part is dissipated. This dissipation occurs in the form of permanent deformation, heat release, cracking or crack propagation. Therefore, to minimize rutting and its negative effect, the part of dissipated work in each loading cycle should be minimized. The work dissipated per loading cycle when a constant stress is applied can be expressed as follows:

$$W_c = \pi\sigma_0^2[\frac{1}{G*/\sin\delta}] \quad (1.1)$$

Where

W_c , σ, and G* are the work dissipated per load cycle, stress applied during load cycle, and complex modulus, respectively.

1.10 Summary

In this chapter different research studies that attempted and tried to address different aspects of cold mixtures and particularly, effect of rejuvenators with respect to cold mixtures are discussed. Firstly, the concept of cold recycling described, and cold mixture variability explained along with the definitions. Subsequently, cold mixture's structure and ingredients including bitumen as a key ingredient and its chemical bonding and functional groups were illustrated. Mix design consideration and its corresponding parameters such as curing time, compaction and temperature were illustrated along with corresponding research findings in this area. Then, conventional, and possible additives that are being used in this filed were introduced and discussed. Finally, rejuvenators as promising additives to rehabilitate the aged binder performance in the cold recycled materials were introduced. Rarely found data in this field confirmed the significant promising effect of rejuvenators on the increased performance of cold mixes. However, there is still little information about several parameters in the field of cold recycling and rejuvenators employment to increase the asphalt payments rheological performance. The absence of comprehensive knowledge makes it to be an interesting topic for lots of research attempts to find out the effect and subsequently optimize its usage in the field pavements. Having precise understanding about the impact of rejuvenators on the performance improvement of cold recycling mixes and its ingredients including RAP and its bitumen content is one of the areas that more studies are needed. Specifically, the quantification of the rejuvenator addition impact in parallel to different mix preparation protocols (i.e., temperature, compaction, RAP size and RAP binder grade, etc.) need to be studied. Finally, yet importantly it needs to be clarified whether the rejuvenators impact origins in the chemical reactions between rejuvenators and aged bitumen or it has roots in the physical impacts (such as reduction in effective viscosity). Such information is of great importance for future alteration of rejuvenators and having optimized and sustainable cold recycling pavements with minimum amount of bitumen usage during the process.

CHAPTER 2

METHODOLOGY

This chapter of the thesis discusses the structure of the experiments and the reasons behind the methodology to address the predefined objectives. In addition, materials and apparatus used in the experiments of the current study are elaborated in this chapter.

2.1 Experimental Plan

The experimental tests in this thesis were divided in two main subsections to evaluate the effect of rejuvenators on cold recycled asphalt mixes in different aspect, including (Figure 2.1):

1. Effect of rejuvenators on the cold mix.
2. Effect of rejuvenators on the extracted bitumen of the cold mix.

The work done on cold mixes is concentrated on the rejuvenator's effect on the cluster creation in the mixes and rejuvenator's effect on the tensile strength of cold mixes. The part on the bitumen looks at rejuvenator's effect on the thermomechanical properties of the aged binder and its possible chemical impact on the RAP binder. Both parts are explained in the next sections.

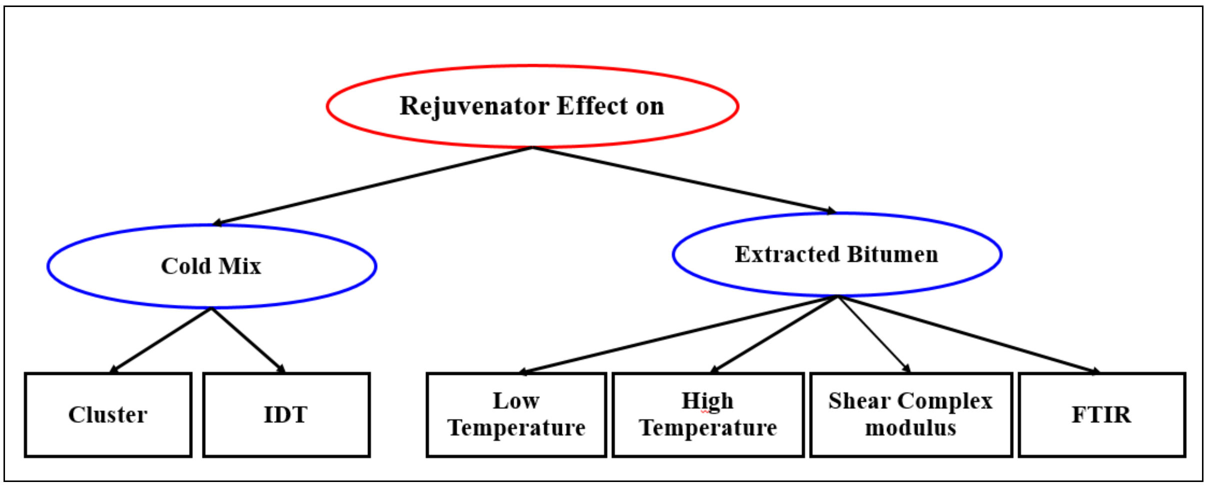


Figure 2.1 General holistic structure of experiments

2.1.1 Cluster Test

Having a new cluster in the mix is an indicator of the fact that the asphalt particles are stacked together which is basically is done by virgin bitumen as binder in the mix. Therefore, cluster creation in the mix as a result of rejuvenators in the mix can be considered as a positive contribution of the rejuvenators to CRM.

. Initially, to identify the effect of the rejuvenator on cold mixes, the possible contribution of the additives on the formation of new cluster in the mix was evaluated. This was done using a sieving method to distinguish the initial fine particles from the formed cluster in the mix. This method of cluster creation quantification was developed by (Bressi et al. 2015). The overall structure of clustering tests on different specimens as well as the initial evaluation of the materials used in the tests are provided in Figure 2.2 where the following considerations were regarded in each case (the same sequence of I, II and III are pictured in the Figure):

I. Virgin aggregates were one sieve size bigger than RAP to determined cluster creation after mixing materials and sieving.

II. Compaction was done by SGC machine for specimens with 100 mm diameter using 200 Gyrations.

III. Different amounts of rejuvenator content were considered to see the effect of rejuvenator dosage on the cluster formation on loose mixtures.

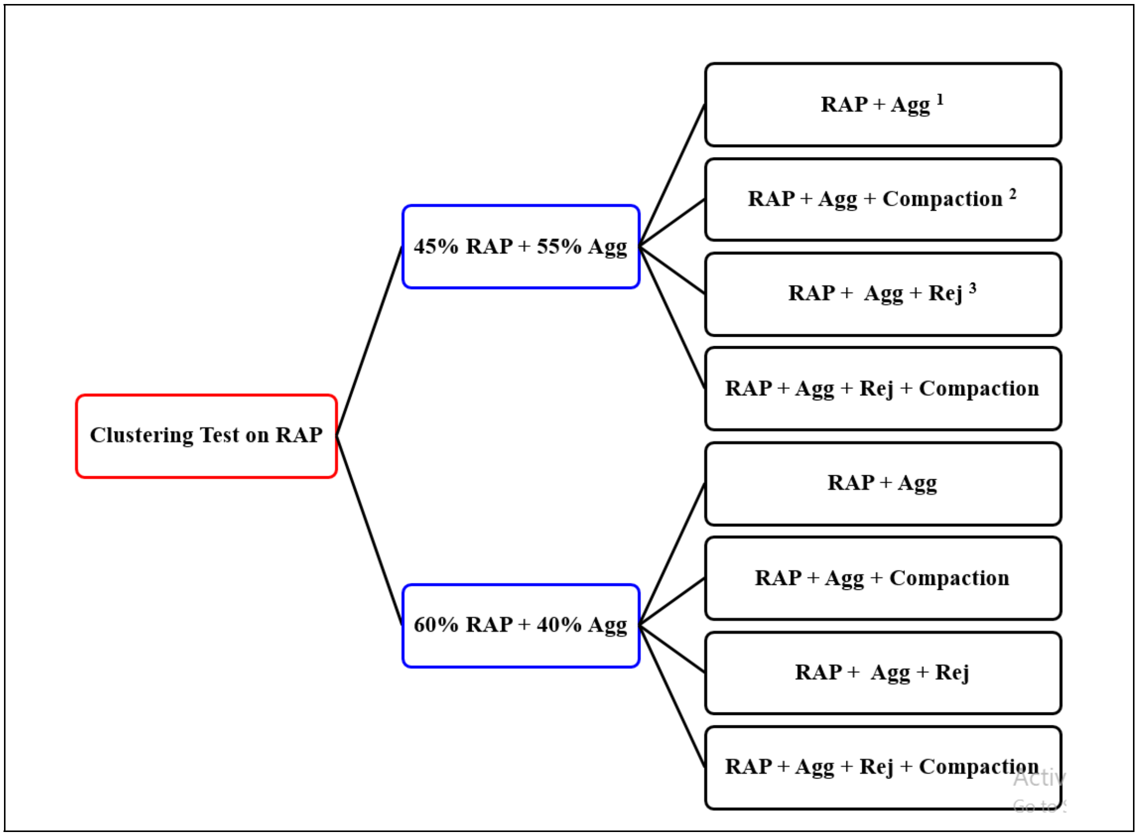


Figure 2.2 Clustering test structure.

In this test, four main variables including RAP content, rejuvenator dosage, compaction and curing time were investigated through a series of tests as illustrated in Table 2.1. To evaluate the effect of rejuvenators on the clustering of the RAP, the specimens were made by mixed of RAP, virgin aggregate, and rejuvenator. Figure 2.3 provides an overview of the specimen preparation for the current investigation. It is important to note that compaction was used on part of the specimens. This was done because we believed that the mechanical energy of the compaction may have an impact on the rejuvenator dispersion in the specimens, so it should increase the number of clusters even if the compacted specimens are broken apart afterward.

Table 2.1 Clustering test experiment plan

Variables	RAP %	Rej %	Compaction		Curing time
			Compacted	No Compaction	
Series of Test	45	0	✓ (2 rep)	✓ (2 rep)	2 Weeks
					4 Weeks
					6 Weeks
		7	✓ (2 rep)	✓ (2 rep)	2 Weeks
					4 Weeks
					6 Weeks
		10	✓ (2 rep)	-	2 Weeks
					4 Weeks
					6 Weeks
		12	✓ (2 rep)	✓ (2 rep)	2 Weeks
					4 Weeks
					6 Weeks
	60	0	✓ (2 rep)	✓ (2 rep)	2 Weeks
					4 Weeks
					6 Weeks
		7	✓ (2 rep)	✓ (2 rep)	2 Weeks
					4 Weeks
					6 Weeks
		10	✓ (2 rep)	-	2 Weeks
					4 Weeks
					6 Weeks
		12	✓ (2 rep)	✓ (2 rep)	2 Weeks
					4 Weeks
					6 Weeks
Total number of specimens: 28					

In the mixes used for the clustering tests, RAP was the fine part, and the virgin aggregates were the coarse part. To do so, a bigger size of aggregate fractions was used in the mix so that the fine and coarse part are easily distinguishable using an intermediate sieve. The amount of aggregate in each specimen was determined considering the following equation:

$$W = Dmax \times C1 \tag{2.1}$$

Where W represents the weight of the upper fraction [g], Dmax represents the diameter of the upper fraction (virgin aggregate), and C1 is a constant factor. Subsequently in each case the RAP was added to have a mix with 45% and 60% RAP. Two different fractions of 0.08/0.630 and 1.25/2.5 were used in the test to investigate the effect of size of fraction and amount of bitumen (bitumen content in small size is more than bigger one) in cluster creation. For the bigger size of the RAP (i.e., 1.25/2.5) C1 was 150, according to Oreskovic et al. (Orešković, Bressi, Di Mino, & Presti, 2017). While C1 =300 was considered for the specimens with RAP 0.08/0.0630 mm. This was because of the limitation of the weight of the specimens for the compaction device (minimum value of 500 g).

To find out the optimum dosage of the rejuvenator within the mix, different amounts of additives (rejuvenators) with the dosages of (0%, 7%, 10%, 12%) were considered for the specimens preparation (these values were selected after pre-investigation tests, available information in the literature and suggested dosage by the manufacturer of the commercial rejuvenator used in the study).

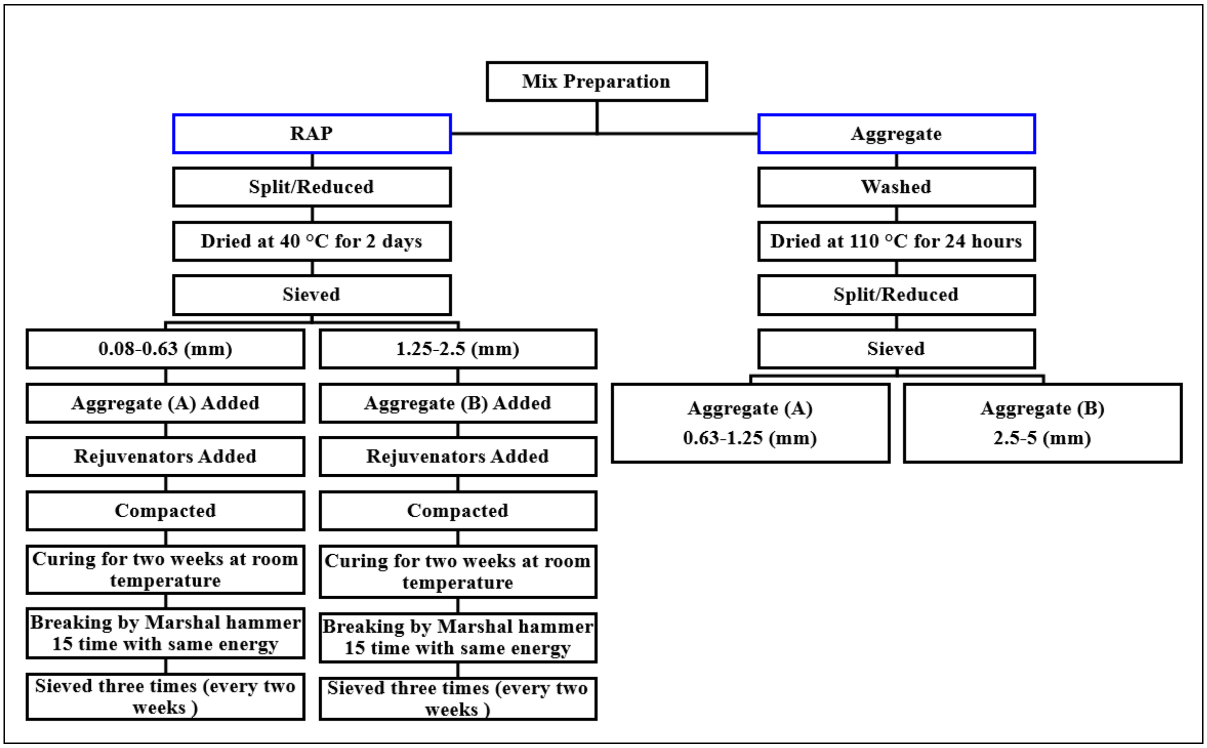


Figure 2.3 Cold mix preparation for clustering tests

In addition, to quantify and isolate the effect of mechanical force on the clustering (during the compaction), specimens with and without compaction were prepared in each case of mixes. The RAP was put in the oven and dried for at least 48 hours before the mixing process.

The compaction was conducted using the gyratory compaction approach. The gyratory compaction approach, made with a shear gyratory compactor, SGC (Figure2.4), physically simulates the compaction of different layers of the pavement under the roller in the road pavement practice. Besides, it also imitates the traffic loading on the pavement.

Figure 2.4 Shear Gyratory Compactor

Compaction improves the interlock between the particles in the mix. This is done using two cylindrical plates on top and bottom of the mould, so that the load is distributed evenly on the specimen. Moreover, having the specimen properties including maximum theoretical relative density, and mass of the mix in the mould, the air voids in the specimen can be monitored during the compaction. SGC compaction specification in this study were as follows:

- Central axis angle by the vertical axis (angle of gyration): 1.25°
- Upper plate pressure: 600 kPa
- Gyration rate: 30 rpm
- Specimen diameter: 150mm

It should be mentioned that, due to the compacting problem for the specimens with RAP but without rejuvenator, the mix and mould in all cases were heated to a specific predefined temperature (40 °C) before compaction. Without this increase in temperature, the cohesion was too low with mixes without rejuvenator (i.e mix with only RAP and aggregate) to have solid specimens (Figure 2.5).

(a) (b)

Figure 2.5 SGC compacted specimens: a) RAP, and b) RAP+Rej

After compaction, all specimens had 2 weeks of curing time at room temperature (Figure 2.6). All the specimens were then broken apart using marshal hammer with 15 hits with same energy (Figure 2.7). This was done by putting specimens on the side to make sure that we do not replicate the compaction effect. It should be mentioned that is possible that some clusters created during the compaction are destroyed during the hits with the Marshal Hammer. Therefore, results are not absolute, they are valuable for comparisons between specimens but they may not represent the true % of new clusters.

Figure 2.6 Cured compacted samples

Figure 2.7 marshal hammer for breaking samples

2.1.2 Cluster creation percentage calculation

In the study method of cluster creation quantification developed by (Bressi et al. 2015) was employed to identify the percentage of cluster creation in the mix. Once the mix passed curing time, it was sieved again with the sieve. Part of the RAP was retained by the sieve (while before the mixing zero percentage of the RAP was retained) because the RAP activated binder within the RAP was adhering to other RAP and aggregated particles, creating clusters that could not pass through the sieve (Figure 2.8). This is also why the virgin aggregate fraction just above that of the RAP was chosen so that to help distinguish the new cluster. Indeed, as soon as the RAP particles started to stick together they did not pass through the sieve and were retained in the upper fraction. The difference between the amount of the not sieved material (including virgin aggregate and stacked RAP particle) and initial virgin aggregate is equal to the amount of the RAP content that contributed to the cluster creation. Dividing the amount of this difference by the initial RAP content weight results in the amount of cluster creation percentage used in this study. Figure 2.8 shows an example of calculation for a sample with 60% RAP content.

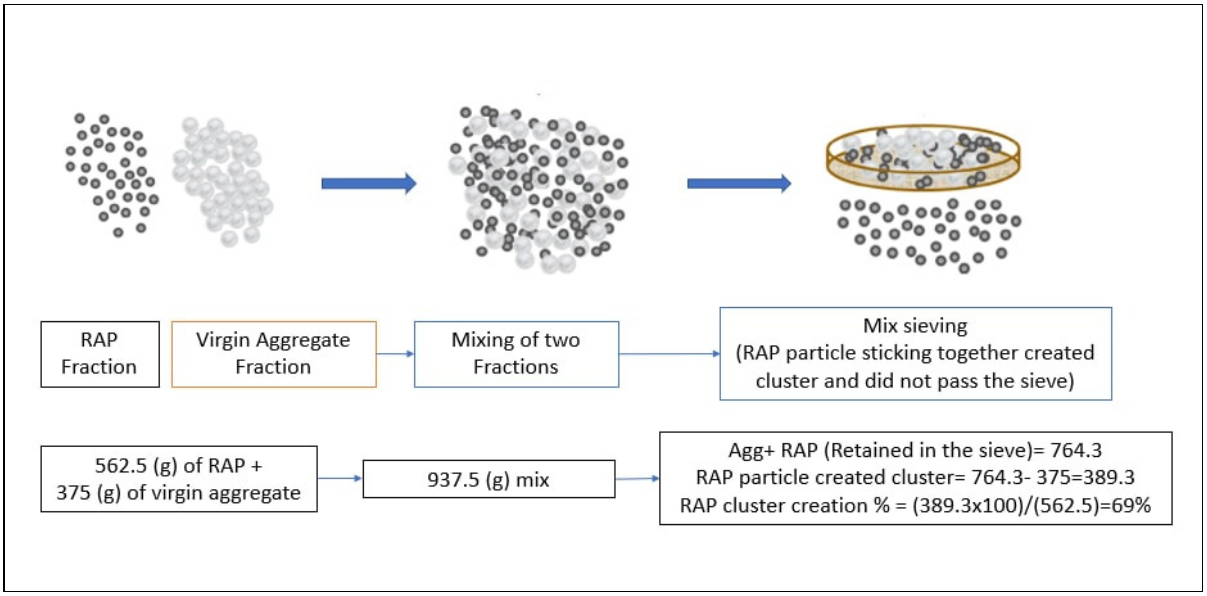


Figure 2.8. Schematic view of cluster creation identification in the mix
Adopted from (Bressi et al. 2015)

2.1.3 IDT (indirect tensile test) IDT TEST

The indirect tensile strength tests are considered as one of the most useful methods to evaluate the cohesion, the tensile strength of asphalt mixes. In this part of the research, the objective was to determine the indirect tensile (IDT) strength of RAP, with and without adding rejuvenator on cold mix material. In this test all specimens had 100% RAP content. It was not possible to compact the specimens without rejuvenators at room temperatures, therefore, to unify the test and also to investigate the warm preparation temperature effect on the IDT tests two temperatures of 40 °C and 60 °C were considered for the test plan. Table 2.2 illustrates the holistic test structure used in the IDT evaluation.

Table 2.2 IDT test structures and samples

Variables	Temperature	Rejuvenator content
Series of Test (100% RAP)	40 °C ✓ (2 repetitions)	0%
		2%
		4%
		7%
		10%
		12%
	60 °C ✓ (2 repetitions)	0%
		2%
		4%
		7%
		10%
		12%
Total number of specimens: 24		

In this test, the weight of the specimens was calculated based on standard ASTM designation D6931-17. To this end the maximum specific gravity of RAP and mix with rejuvenator should be known. Based on standard ASTM D2041/D2041M-19 the maximum gravity for RAP and mix (RAP+2%, 4%, 7%, 10% and 12% of rejuvenator content) were calculated as 2.584, 2.578, 2.563, 2.541, 2.531, 2.524, respectively. Besides, direct experimental measurement showed the same value, confirming the calculation. The target air voids level was 5.0 %, but all the compacted specimens used in this study ended up with air voids of 4.0 ± 0.5%.

There were 24 specimens in total (2 of each mix). Before compaction, all materials were heated to 40 ℃ and 60 ℃, (because of compacting problem for RAP without rejuvenators). First, RAP and mould were heated up to 40 ℃ (Figure 2.9) then RAP was mixed with Rej (mixed one minute by hand and 1 minutes and 9 seconds by mixer according to the Bressi et al (2015) study and available standars for mixing processes). Afterward samples were put back on the oven to reach the same temperature (around 40 ℃ ± 3℃) before compacting by SGC. Two replicates for each combination of the testing conditions were also used. For all samples, the tests were repeated at 60 ℃ to quantify the effect of preheating temperature on the IDT test results. After compaction, the specimens were put in bowls to cool down before measuring the height with a digital caliper at three locations on the specimens (Figure 2.10).

Figure 2.9 Heating mix and mould in the oven before compaction

Figure 2.10 Measuring the dimension of cured samples

Table 2.3 IDT test mixture condition

Variable (test condition)	value
Test Temperature (℃)	60℃ and 40℃
Loading Rate= deformation /time	~50 mm/min
Weight of samples (kg)	~3 kg
Height (mm)	150mm
Gyrations	200
Replicates	2

2.1.4 Extracted binder characterization

To identify the effect of the rejuvenator on the characteristics of the binder content in the RAP, four groups of standard tests were conducted. This set of evaluation encompasses low temperature, high temperature , shear complex modulus and FTIR examination (on the extracted bitumen from mix with different percentages of rejuvenator dosage as shown in Figure 2.11).

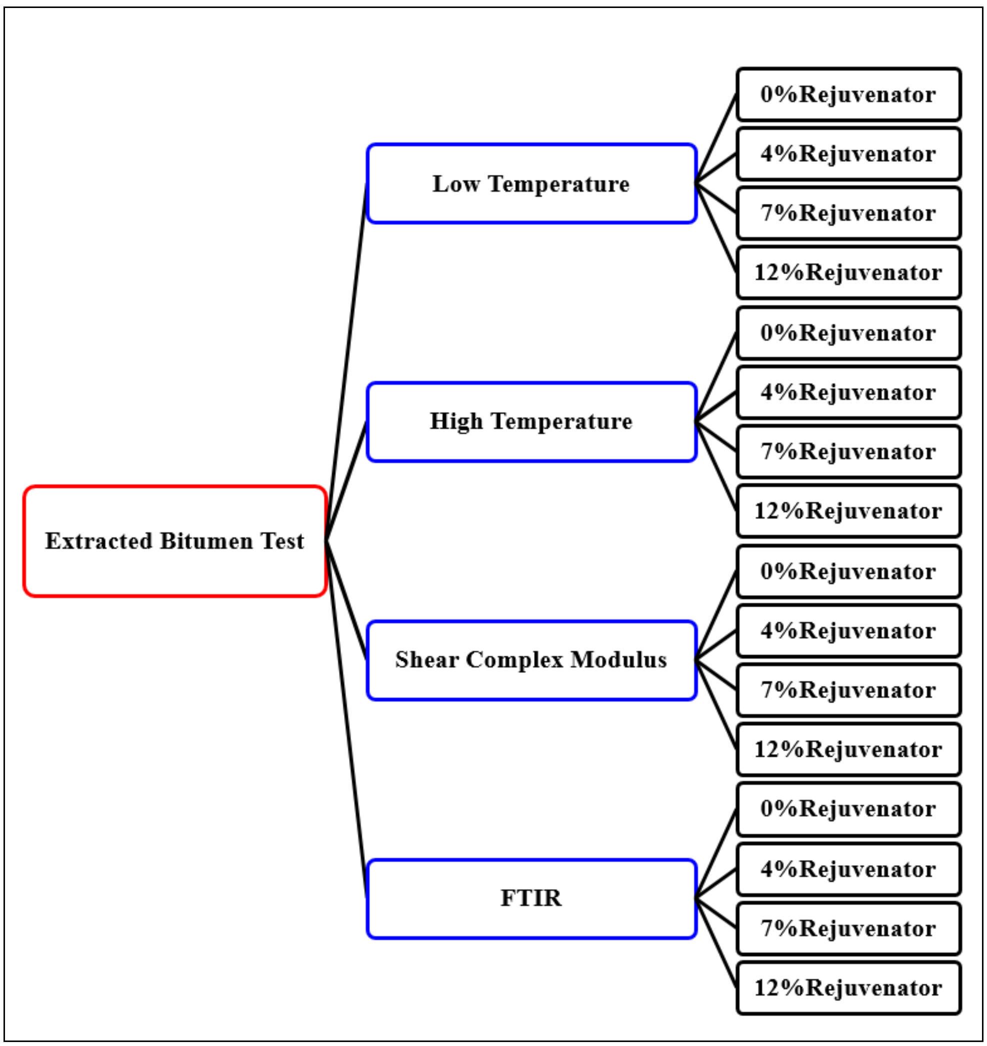


Figure 2.11 Extracted binder characterization test for mix with different dosages of rejuvenators.

2.1.4.1 Low temperature – Bending Beam Rheometer (BBR)

This test measures the low temperature stiffness and relaxation of binder in function of time to determine the resistant of binder to low temperature cracking. In the BBR test, small beams of bitumen (Figure 2.12) is kept on an alcohol bath for one hour before applying a constant load at the centre of the specimen by the machine. The deflection is measured against the time and the stiffness and the change in stiffness, the relaxation, are also calculated. The BBR temperature is calculated based on both the stiffness and the change of stiffness. The low temperature grade is the minimum temperature ate which both of the following criteria are met:

a. BBR stiffness values < 300 MPa, and > 300 MPa

b. BBR m-values < 0.300, and > 0.300

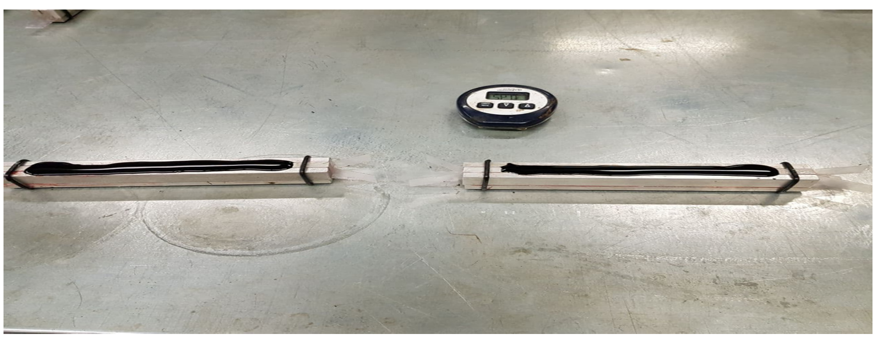


Figure 2.12 Bitumen Beams for BBR test

2.1.4.2 High temperature – Dynamic Shear Rheometer (DSR)

To characterize and evaluate the viscous and elastic behaviour of asphalt binders the dynamic shear rheometer (DSR) is used in the range of medium to high temperatures (Figure 2.13). Results of the characterizations are used in the Superpave PG asphalt binder specification. In this section, according to other Superpave binder tests, the actual temperatures predicted in the area where the asphalt binder was placed, determine the test temperatures used in the experiments.

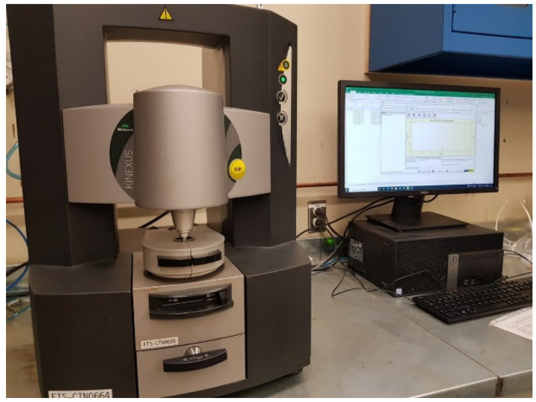


Figure 2.13 Dynamic shear rheometer.

DSR tests basically use a thin layer of asphalt binder that is sandwiched between two circular plates where, the lower plate is fixed while the upper plate oscillates back and forth across the sample at 10 rad/sec (1.59 Hz). Figure 2.14 shows the specimens used in the experimentations. It should be mentioned that the asphalt binder specimens used in the DSR tests were on three states of unaged, RTFO aged, and PAV aged. AASHTO T 315 standard was used in this study to determine the rheological properties of asphalt binder in DSR tests.

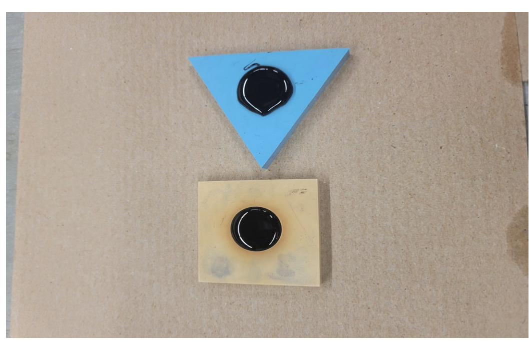


Figure 2.14 DSR Specimen

2.1.4.3 Shear complex modulus

The other test that was carried out for rheological behaviour examination of extracted bitumen from the RAP mix rehabilitated by different dosages of rejuvenator (see Figure 3.15) is a complex shear modulus test. This test was conducted using the DSR machine and the AASHTO-T-315 was adapted for the analysis. Results were analyzed both on the dynamic shear modulus and phase angle of asphalt binder in a oscillatory shear condition. The test was applicable to asphalt with dynamic shear modulus values between 100 Pa to 10 MPa and which can usually be obtained for the modulus between 6° C and 88 ° C at an angular frequency of 10 rad/s. To investigate the results, the measured shear modulus responses (G*) of the bitumen were first modelled with 2S2P1D model which in turn is a generalization of the Huet-Sayegh model (Olard & Di Benedetto, 2003). In this model, seven constants should be determined at the test temperature (See Figure 3.15). Then the G* can be determined using Olard and Di Benedetto formulation as follows (Olard & Di Benedetto, 2003):

$$E^*_{i\omega t}=E_{00}+(E_0-E_{00})/(1+\delta(i\omega\tau)^{-k}+(i\omega\tau)^{-h}-((\boldsymbol{i\omega}\beta\boldsymbol{\tau})^{-1}) \qquad (2.2)$$

In this equation *h* and *k* are exponents, where $0 < k < h < 1$. E_{00} represents the static modulus when $\omega\tau \rightarrow 0$ (i.e., low frequencies and high temperatures) with $\omega = 2\pi$ frequency while E_0 is the glassy modulus when $\omega\tau \rightarrow \infty$ (i.e., high frequencies and low temperatures). It should also be mentioned that the term (E0 − E00) βτ is called the Newtonian viscosity shown by η in Figure 2.14.

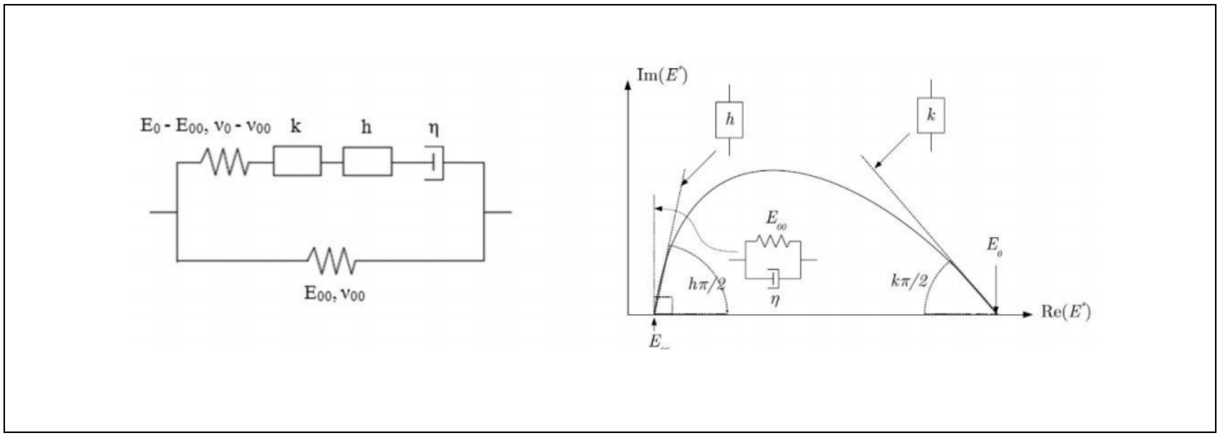


Figure 2.15 2S2P1D schematic model used for the shear complex modulus
Taken from Saliani, Carter, Baaj, & Mikhailenko (2019).

2.1.4.4 FTIR

Fourier-transform infrared spectroscopy (FTIR) has been widely used for the quantitative and/or qualitative analysis of bitumen in the asphalt technology studies (Weigel & Stephan, 2017) (Karlsson & Isacsson, 2003) (Weigel & Stephan, 2017). FTIR allows the identification and quantification of functional groups in the bitumen, and it's a very effective method for investigation on the characterization of binder chemical composition (Karlsson & Isacsson, 2003) and aging behaviour of the RAP binder (Yang, You, & Mills-Beale, 2015). This technique works based on the difference of the absorbance of infrared light by covalent bonds in molecules. This difference on the absorption of different types of bonds which is distensible by the intensity and frequency of the absorbed light enables the identification of chemical functionalities. It should be mentioned that the FTIR analysis in the case of bitumen characterization requires caution since there may be many sources of interferences (such as overlapping of different absorbing bonds, shift of wave number, structural geometry of investigated functional group, etc.).

Figure 2.17 shows a typical FTIR spectra and important functional group of bitumen and a commercial rejuvenator (bio binder). The peak height and band area in FTIR analyses are generally used to identify and indicate the concentration of certain bonds in the material.

For instance, bands for indexes like carbonyl (C=O) and sulfoxide (S=O) are used to characterize the oxidation in the asphalt binder (also referred as oxygenation indexes (Weigel & Stephan, 2017). Other bonds indexes like aromaticity index (AR), C-O bond index, Alkanes including CH_2 bond index and CH_3 bond index have been also used to investigate the effect of aging on the bond changes (Yang et al., 2015). These index provides reliable data about the aliphaticity, aromaticity and oxygenation rate of the bitumen (Feng, Bian, Li, & Yu, 2016).

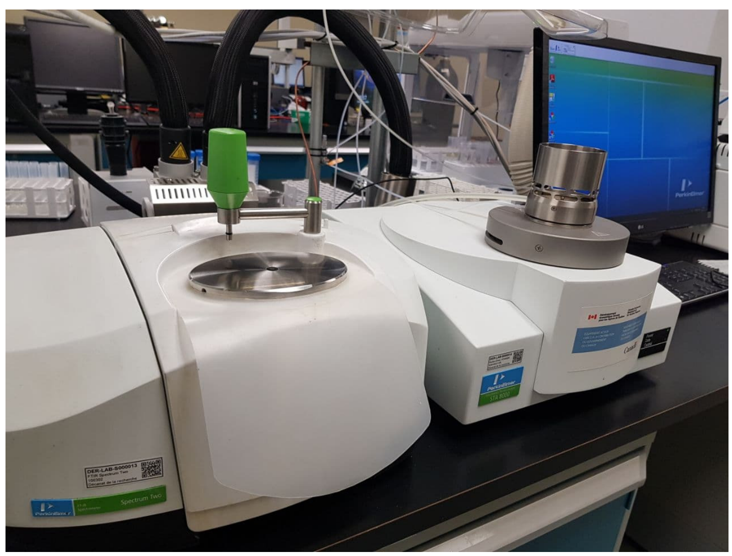


Figure 2.16 FTIR test machine

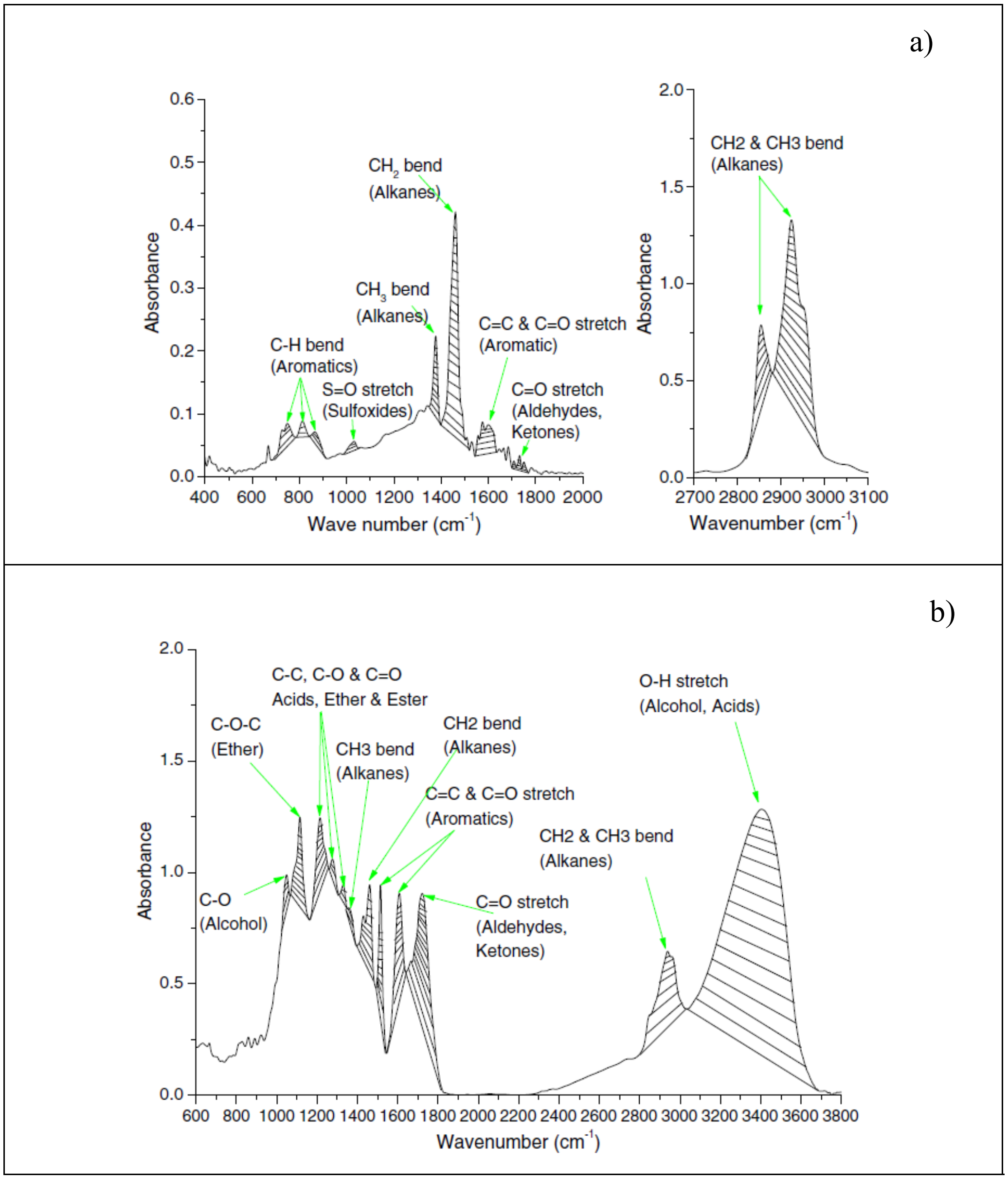


Figure 2.17 The FTIR analysis for a) bitumen and b) bio-binder

Taken from Xu Yang (2014)

Four kind of specimens including virgin bitumen, aged bitumen extracted from RAP and, modified bitumen extracted from the mix of RAP, rejuvenator, and pure rejuvenator were used to identify the oxidation process and the contribution of rejuvenator on the chemical bonding of the rehabilitated binder (Figure 2.18). The spectra between 4000 to 400 cm^{-1} at a resolution of 4 cm^{-1} (suggested for bitumen studies (Larsen, Alessandrini, Bosch, & Cortizo, 2009)) was recorded and the IR spectral bands were analyzed via software included i#n the FTIR testing machine. Six trials were considered for each sample at a given condition.

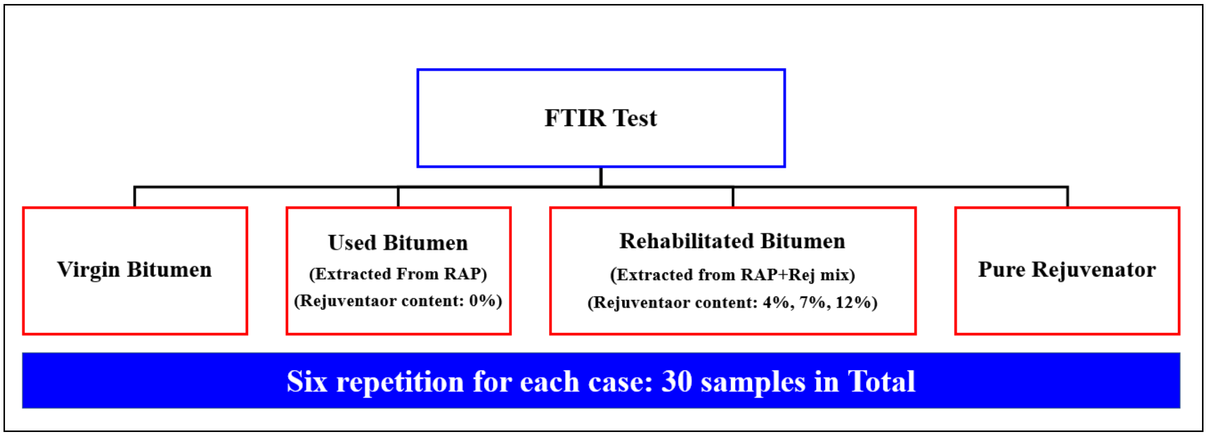


Figure 2.18 FTIR test structures and samples

2.2 Materials

This section describes the different materials that were used in this study. A single source of Reclaimed Asphalt Pavement (RAP), a single source of virgin aggregate and a rejuvenator was used.

2.2.1 Reclaimed Asphalt Pavement - RAP

RAP as the structural body of all specimens in the present study was the most important ingredients used in the experiments. RAP used for this study was collected from DJL-Carignan. The collected RAP materials had been mixed and consequently had been split or reduced. The general evaluations made on the RAP includes are illustrated in Figure 2.19.

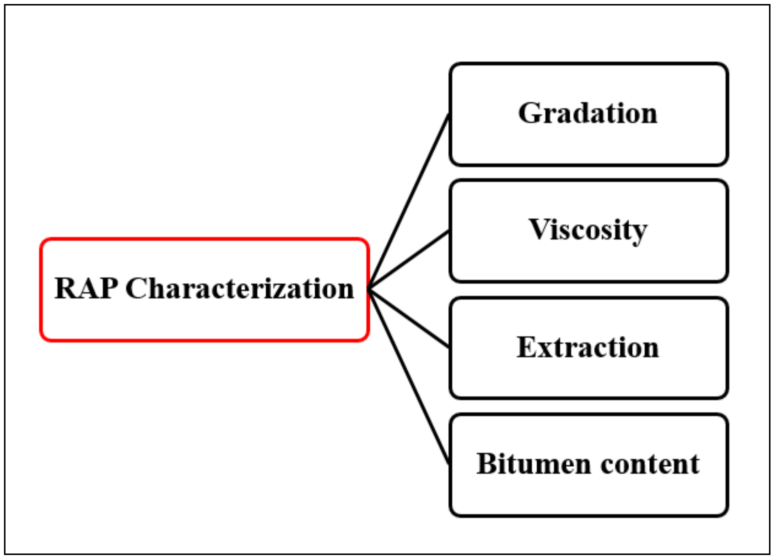


Figure 2.19 RAP evaluation process

The RAP was dried at 40 ºC before sieving according to ASTM D448-03 standard. This was done to achieve the sieving analysis of the investigated RAP including nominal maximum particle size and gradation. The corresponding gradation of the RAP and virgin aggregate is depicted in Figure 2.20. The bitumen content of RAP was determined according to ASTM D6307 standard. Table 2.4 provides the RAP characteristic after evaluation. It should be mentioned that to use the materials, they were evaluated and measured based on their gradation, extraction, viscosity, softness point, penetration, high temperature, ignition oven, density and low temperature.

In order to address the clustering effect test, RAP was sieved into 5 sizes (including the minimum size of 0.0/0.08 which encompass the filler according to the ASTM C136-06 standard. Details of the 5 sieve sizes of RAP is provided in Table 2.5.

The fractions that were used during the clustering experiments were 1.25/2.5mm and 0.63/0.08. That was to understand the effect of bitumen content on the specimen on the cluster creation.

Table 2.4 Characteristics of the RAP

Properties	Measurements
Bitumen content, %	5.31
Maximum specific gravity	2.584
Penetration, 0.01mm	9

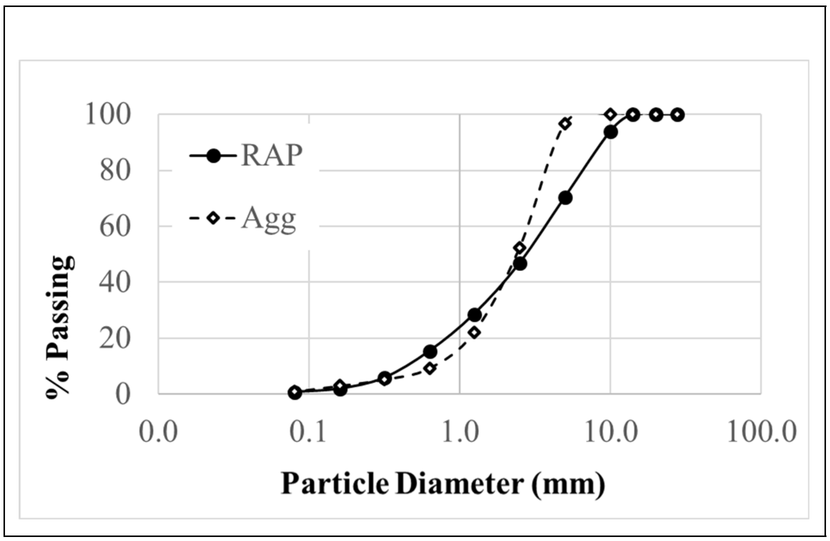


Figure 2.20 Gradation curve : black solid line with round marking is RAP, and white squre dashed line is Aggregate

Table 2.5 Rap fraction and its corresponding bitumen content

Fraction, mm	Fraction Percentage, %	Bitumen Content, %
0.0/0.630	15.3	7.19
0.630/1.25	13.23	6.88
1.25/2.5	18.14	5.48
2.5/5	23.62	4.80
5/10	23.37	3.73

2.2.2 Extracted bitumen

To investigate the effect of rejuvenator on the bitumen content of RAP, bitumen extraction is the first step, see in figure 2.21. To this end, 4 kind of mix samples (RAP+Rej) (with 0% , 4%, 7%, 12% Rej) were extracted. To achieve enough amount of bitumen from each kind of specimens, the extraction repeated at least four times. This was done in order to fulfill the requirement of bitumen-related test (FTIR, low and high temperature as well as complex shear modulus tests). All steps of are shown in Figure 2.22.

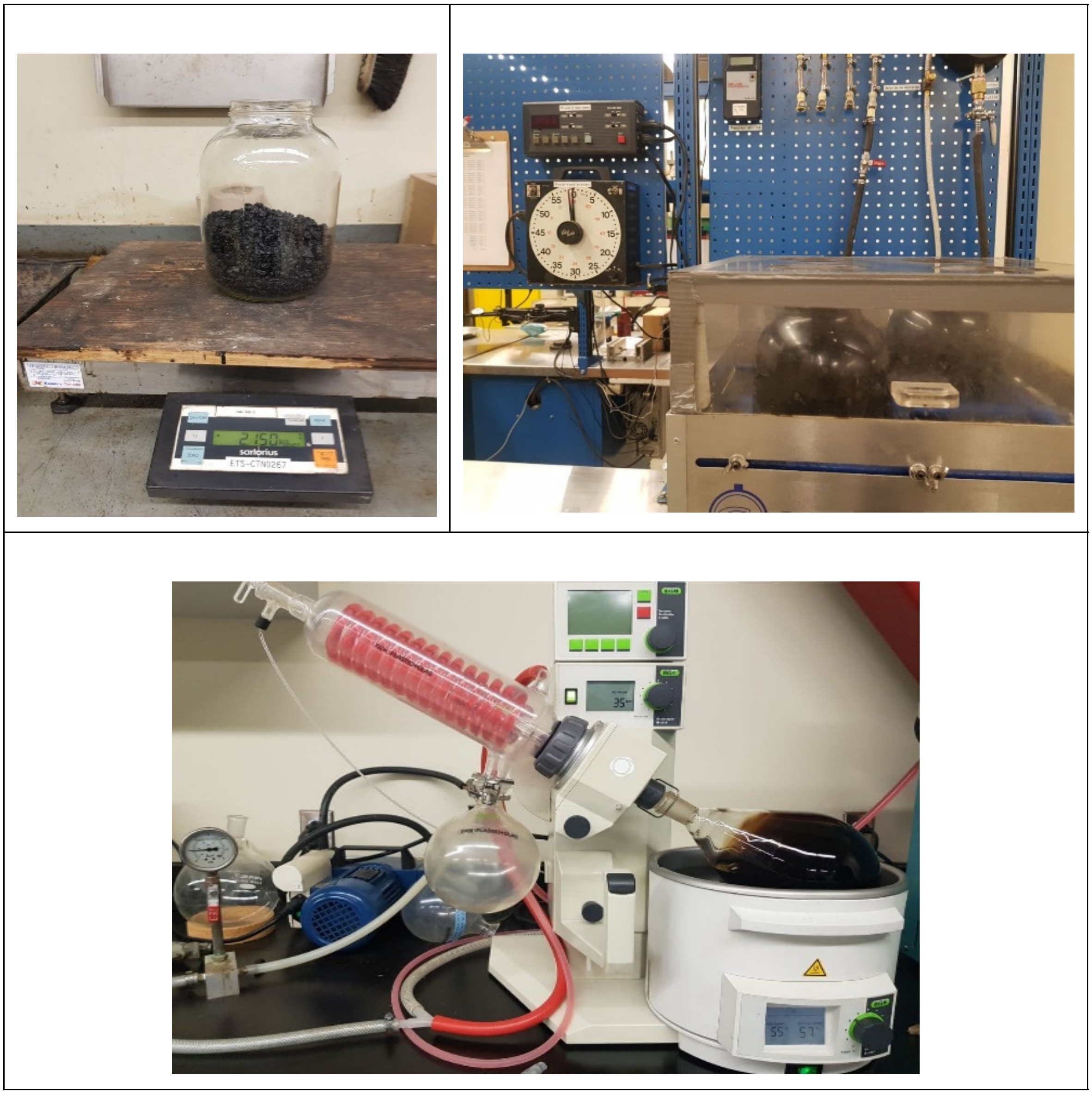


Figure 2.21 Bitumen extraction equipment and process.

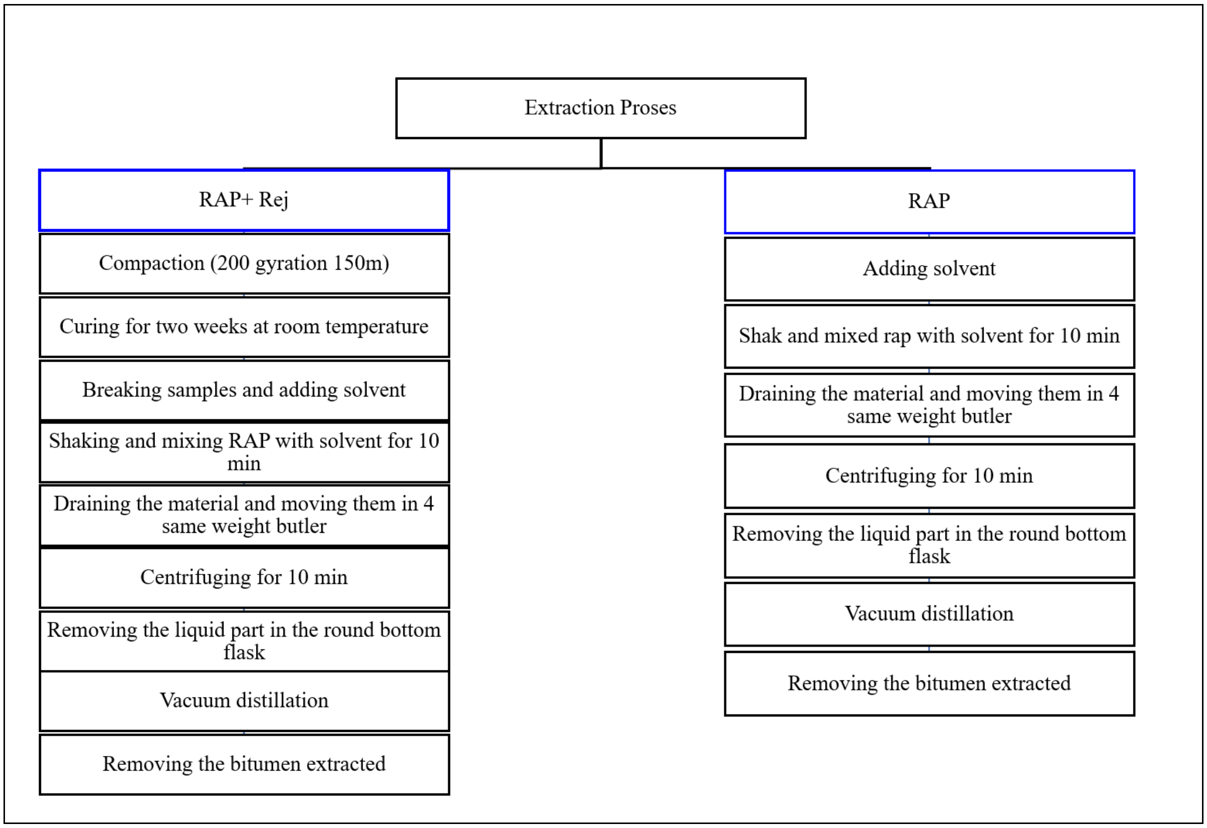


Figure 2.22 Bitumen extraction process

2.2.3 Virgin aggregates

To investigate the clustering effect, a single source of virgin aggregates was used as the coarse size. The collected virgin aggregates were mixed then split to reduce them, then washed to remove very fine particles. Consequently, those materials had been dried at 110 ºC and sieved as according to the procedure taken for the RAP. Details of the aggregate gradation for the clustering test is provided in table 2.6. It should be mentioned that the only fractions used during this research were 2.5/5 mm and 1.25/0.63.

Table 2.6 Gradation details of the virgin aggregate

Fraction	0.0/0.08	0.08/0.16	0.16/0.315	0.315/0.630	0.630/1.25	1.25/2.5	2.5/5	5/10
Weight %	0.92	2.2	2.28	3.9	11.84	30.23	43.2	3.49

Figure 2.23 ASTM standard designation C 136-06 test method for sieve analysis of fine and coarse aggregate

Taken from CIVILBLOG.ORG

2.2.4 Rejuvenator

The rejuvenator used in this study is a liquid additive called Sylvaroad RP1000. This bio-based rejuvenator driven from crude tall oil (CTO) according to the manufacturer was produced to provide high-end reuse of RAP in HMA. The properties of this commercial bio-based rejuvenator including (dynamic viscosity, flash point and density), are shown in table 2.7. According to the producer (Karton inc.), a dosage of 5% could improve the aged binder by 2 sizes and enables homogenous mixing. It has been reported that after 2nd cycle of recycling, rejuvenated mixes with 50% RAP have aging similar to control mix with 0% RAP was identified (ref).

Table 2.7 Properties of the Sylvaroad rejuvenator
Taken from Karton, inc.

Properties	Values provided
Vicosity at 20°C	0.927 Pa.S
Cloud point	<-25 °C
Room state	Clear and bright Liquid
Flash point	>280 °C
Chemical classification	Non-hazardous

Figure 2.24 Sylvaroad rejuvenator at Room temperature

CHAPTER 3

RESULTS AND DISCUSSIONS

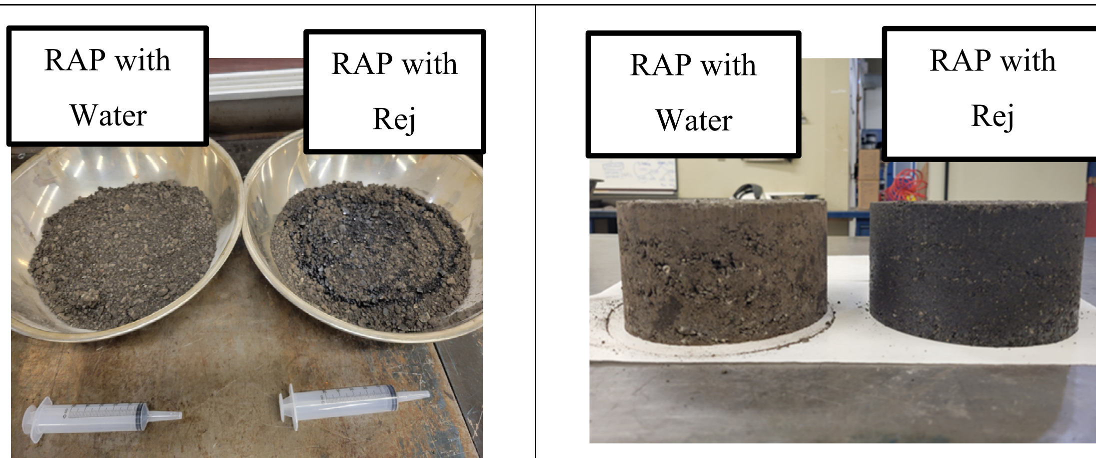
3.1 Introduction

This chapter provides the results concerning the effect of rejuvenator usage on the cold asphalt recycling materials. Results are discussed in the main three subchapters to discuss the effect of rejuvenator on the clustering, on cold asphalt mix rheological properties as well as rejuvenator's effect on the extracted bitumen from the RAP. But lets start with the visual observations of the effect of the addition of the rejuvenator.

3.2 Clustering

3.2.1 Appearance result

Figure 3.1 shows the overall appearance of two cold mixes samples modified by water and rejuvenator as additives (12% additive dosage was considered here). It can be seen that the compacted sample modify by water had more height (in other words less compatibility compared to the rejuvenator-based modification). Besides, it can be seen that the colour appearance in these two samples are completely different showing the strong effect of rejuvenator on the rehabilitation of the bitumen inside the specimen.



(a) (b)

Figure 3.1 Appearance of compariosn of cold mixes samples modified with water and rejuvenator as additives(a) befor compaction ,(b) after compaction

3.2.2 Cluster creation assessment

This part illustrates the effect of amount and size of RAP, amount of rejuvenator, curing time and compaction on the clustering creation in the cold asphalt mixes. To do so, 28 specimens were prepared. Figure 3.2 shows cluster particles in the mixes. (Calculation of cluster creation was mentioned in 2.1.2)

Figure 3.2 Cluster formation and its corresponding particles in the mixes.

- The first and most important variable in this study is the dosage of rejuvenators in different tests. Each of the following variables and their corresponding test results were interpreted according to the presence of different amounts of rejuvenator (0, 7, 10, 12%) in the specimens.
- The next two variables of this research are the effect of amount and size of RAP on the cluttering creation in the mix. To do so, two different contents of RAP including 45% and 60% RAP (which means 55% or 40% virgin aggregate in the mix correspondingly) and two different sizes of 0.08mm and 1.25mm were considered in the preparation of each sample. Results.
- The forth variable is curing time. Here, the final (maximum measured) curing time is 6 weeks for all samples. During this period, each 2 weeks specimens were sieved to track the creation of new cluster. It should be mentioned that in this research after adding additive and compaction, cluster creation was not checked initially.
- The next variable that has been tracked in this test is compaction (by SGC) effect. In this research there are some specimens that were prepared without any compaction to track and estimate the effect of compaction specifically.
- The last, but most important variable in this test, is rejuvenator content effect. In this part the effect of amount of rejuvenator on cluster creation has been tracked. In each specimen, there was 45% or 60% RAP and 55% or 40% virgin aggregate and different amount of rejuvenator (0, 7, 10,12%). Zero percent rejuvenator content sample were prepared to track the effect of other variables (for instance curing, compaction etc.) and consequently subtract their impact on the cases with rejuvenator to estimate the effect of rejuvenator precisely.

Figure 3.3 shows the effect of content and size of RAP on cluster creation. Here all were prepared without any compaction or rejuvenator addition. As it can be seen, there is no difference between the results of all samples during the time and no creation of cluster was observed. Therefore, it can be said that in the absence of rejuvenator or compaction, no new cluster is created (RAP and aggregate).

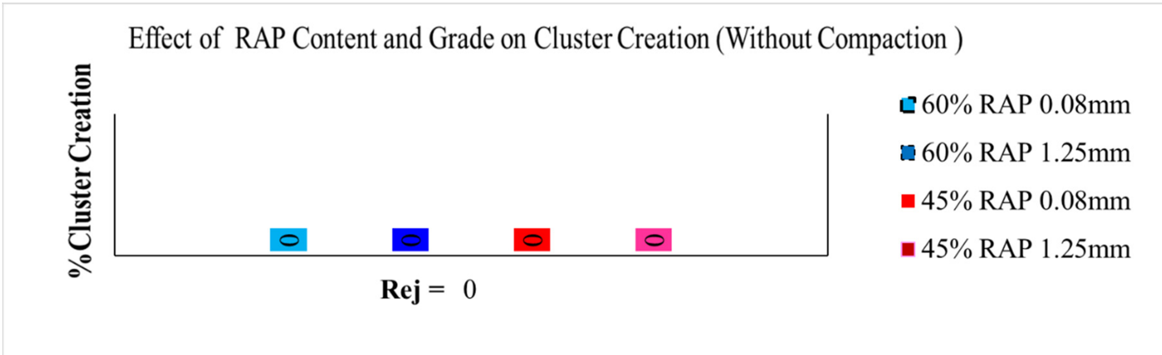


Figure 3.3 Effect of RAP Content and RAP Size on cluster creation

Figure 3.4 show the effect of percentage of rejuvenator on clustering creation on the different amounts and sizes of RAP. Here, to track the effect of rejuvenator on cluster creation, these sample were prepared without compaction.

It can be seen that an increase in the amount of additive lead to increases in the amount of cluster creation. Besides, it can be seen that in each percentage of RAP content samples with higher amount of RAP (60%) had higher cluster creation. Also, in each specific amount of RAP the smaller size of RAP had higher percentage of cluster creation. For instance, it can be seen that the higher amount (higher percentage of RAP) and smaller size of RAP in each amount of additives has the highest amount of cluster creation.

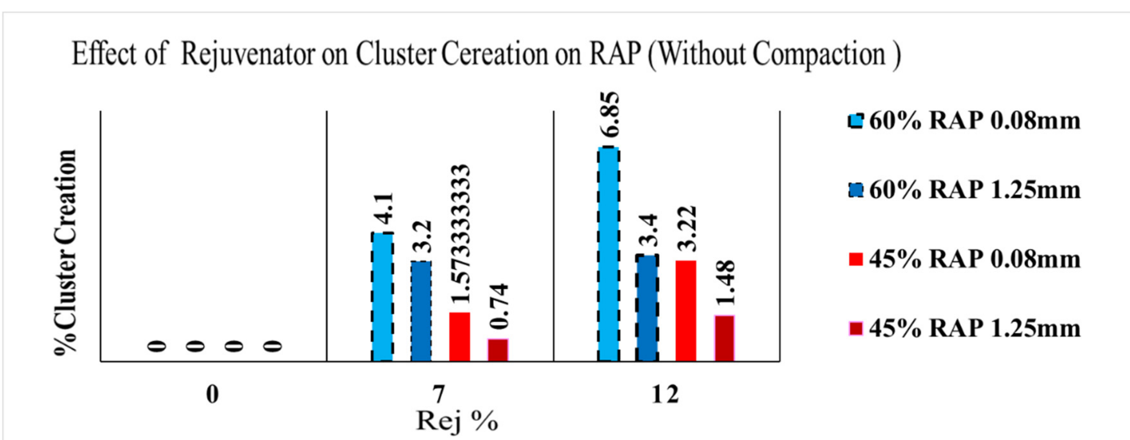


Figure 3.4 Effect of rejuvenator on the cluster creation on RAP(without compaction)

The fact that little to no clustering is observed without compaction is not surprising. Without a mechanical action to force contact between the different particles, only the gravity is pushing the particles together. For that to be enough, higher dosage of rejuvenator seems to be required. However, since RAP stockpiles are big at asphalt plant, higher force are expected, and clustering effect could be observed without compaction.

Figure 3.5 demonstrates the effect of two different contents (60% ,45%) and sizes (1.25, 0.08mm) of compacted RAP on cluster creation in the presence of different rejuvenator content. It is clear that, amount of RAP has a direct effect on the percentage of cluster in presence of other variables. As it can be seen, regardless of RAP size, more RAP led to more cluster creation. Cluster difference between 60% and 45% amount of RAP in both sizes is about 40%.

In terms of size, bitumen in smaller RAP size (0.08mm) has contributed to more cluster formation than bigger one (1.25mm). As it can be seen, even in samples without rejuvenator (Rej%=0) cluster creation in RAP 0.08mm for both amounts (45 % ,60%) is about 10% more than RAP1.25mm. For instance, compacting of a sample with 45% RAP and size of 1.25mm with zero percentage rejuvenator, has negative effect on cluster creation (i.e 1.28% reduction in upper sieve particles or in other word negative cluster), but compacting the specimen with 45% RAP and size of 0.08mm has positive effect on cluster creation (+9.49%). This is because of the fact that the amount of bitumen in the lower size is more than for bigger size. Therefore, ability of sticking and creating the cluster in lower size (0.08mm) is more due to the bitumen content. (See Table 3.5 where the RAP fraction and its corresponding bitumen content are provided).

Figure 3.5 also compares 8 different specimens in terms of percentage of amount of rejuvenator. It is clear that by increasing the percentage of rejuvenator, cluster creation is increased, especially for the compacted specimens. It can be seen that the overall cluster creation on the specimens without compaction are far less than those of compacted specimens. In general overview it can be seen that for the compacted specimens regardless of the size (0.08mm /1.25mm), the tangent of cluster creation curves was reduced by increasing the amount of rejuvenator (in other words the cluster creation is not increasing linearly).On the other hand, the slope of curves for the uncompacted specimens were linear in the range of tested rejuvenator content. However, this behavior can change in higher percentage of additive which were not studied in this research. For the compacted specimens, the rate of increase of cluster creation by adding more amount of rejuvenator is decreased, though it is still positive, therefore it can be concluded that there is an optimum percentage of rejuvenator for each design. Figure 3.3 shows that higher cluster forming belong to the specimens with more mount of RAP (60%) and smaller size of RAP(0.08mm), while, the lower amounts of cluster creation between compacted specimens belongs to the bigger sizes and lower amount of RAP. It should be noted that the amount of RAP is more effective than size of RAP in the matter of cluster creation. Besides, here it is clear that, when there is not any mechanical force (compaction) amount and size of RAP have a positive role in cluster creation.

One interesting point in this figure belongs to the initial state of compacted specimens with 0% rejuvenator where the specimen with 60% RAP (1.25mm) had about around 16% new cluster, while specimen with 45% RAP and same size not only had not new cluster creation, but also shows -2% reduction in cluster. This is because of the fact that in the absence of enough RAP (bitumen inside the RAP) the sticking potential of mix is low to create a new cluster and in the absence of rejuvenator, the compaction process broke some of the initial coarser of aggregated to the smaller size leading to -2% of upper sieve particles observation in the measurements.

The other important outcome of this figure belongs to the point of intersection curves of compacted specimens with different size and same amount of RAP content. For example, adding 7% rejuvenator to the mix with 45%RAP for both sizes of 1.25mm and 0.08mm lead to create new cluster of 23%. To explian this result, we can say, during compaction, aggregate in the specimen with 45%RAP (1.25mm size) have broken and amount of fine part in the specimen was increased which. This in turn tends to create new cluster in the presence of enough additives in the mix. Therefore, compaction by increasing the fine part in the specimen make contributed to a behavior similar to lower size of 0.08mm specimen, and consequently same cluster creation. This phenomenon for specimens 60%RAP happened at 10% rejuvenator where the both size of 0.8mm and 1.25 mm had the same amount of cluster creation of 69.4%.

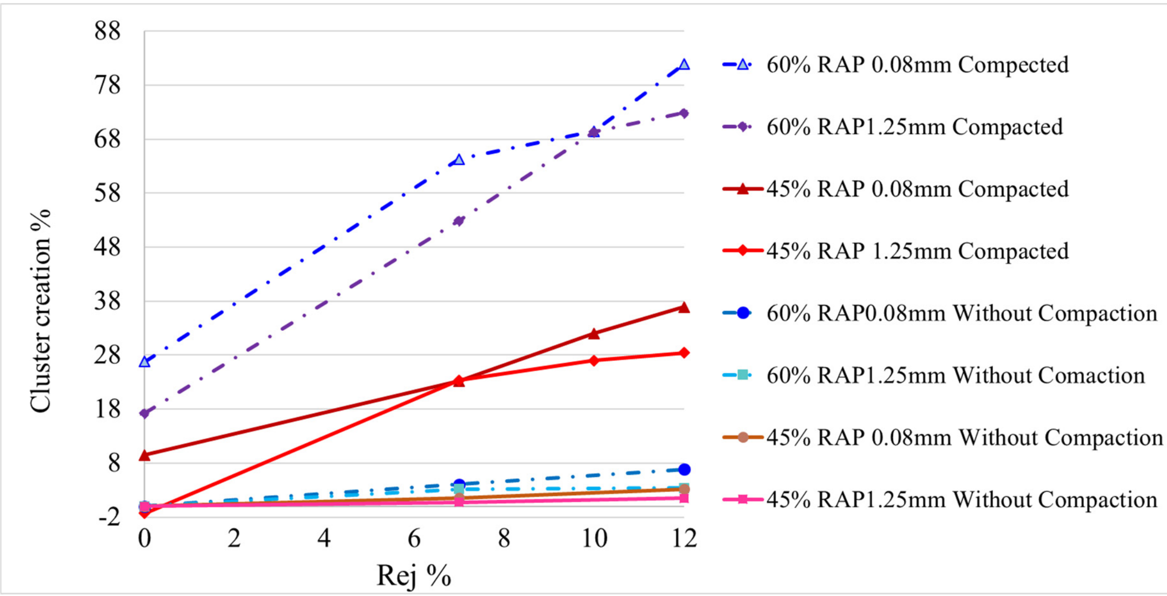


Figure 3.5 Effect of RAP content and RAP size on the cluster formation in the cold mix

Figure 3.6 investigates the details of RAP content and RAP sizes effect on the cluster creation in the specimens without compaction. To have a general overview, the axis range in Figure 3.5 and Figure 3.6 a are same. It can be seen that in the absence of mechanical forces (compaction) specimens with more amount of rejuvenator had more cluster creation. For instance, specimens that had 12% of rejuvenator had almost doubled new cluster creation in comparison with specimens that had 7% rejuvenator. Here total amount of cluster creation after 12% rejuvenator for specimens with 60% RAP and RAP sizes of 0.08mm and 1.25mm are 6.85% and 3.4%, respectively, while specimens with 45% RAP and RAP sizes of 0.08mm and 1.25mm shows 3.22% and 1.48% of cluster creation.

In terms of size the same trend of higher cluster creation for specimens with lower sizes can be found in Figure 3.6. Figure 3.7 Shows the compacted and non-compacted specimens after sieving

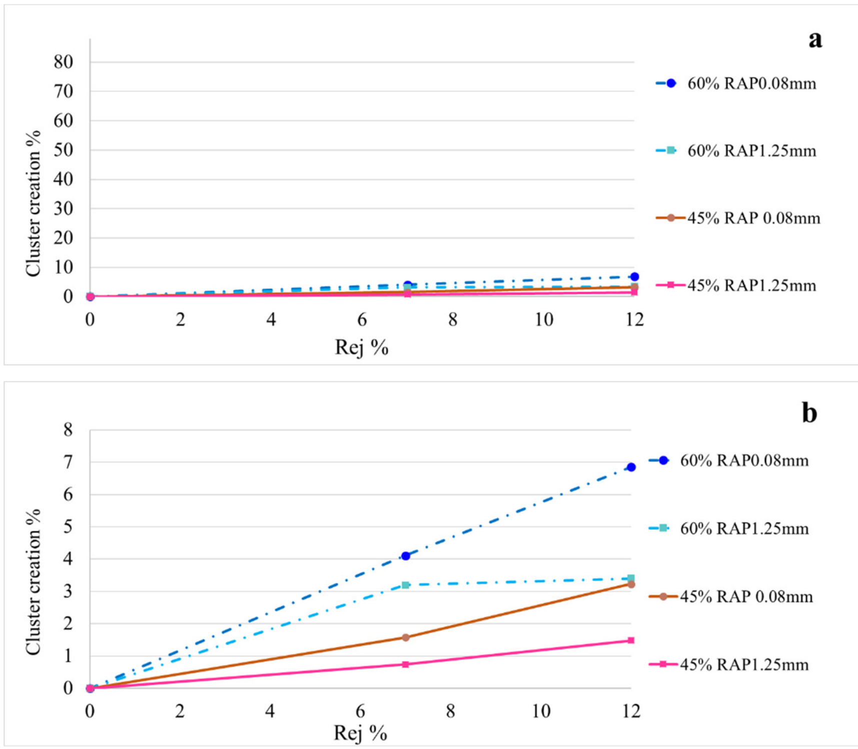


Figure 3.6 Effect of RAP content and size on the cluster formation in the non-compacted cold mix: a) global range of the all tests carried out in this study and b) local rage for this specefi case for compariosn

(a)	(b)

Figure 3.7 Cluster creation of a) uncompacted, and b) compacted specimens

To visualize the effect of RAP content and RAP size on the cluster creation, a comparison of all specimens with different preparation methods (RAP content, RAP size, Rej%, compacted and without compaction) is presented in the column chart of Figure 3.8. It is obvious that, regardless of amount of rejuvenator the highest cluster creation belongs to specimens that contain more amount of RAP in each size.

For example, in the case of specimens with 12% rejuvenator, specimens with 60% RAP in both size (0.08mm, 1.25mm) had more cluster formation than specimens with 45%RAP. This trend can be tracked in all other specimens with or without compaction, including any percentage of rejuvenator.

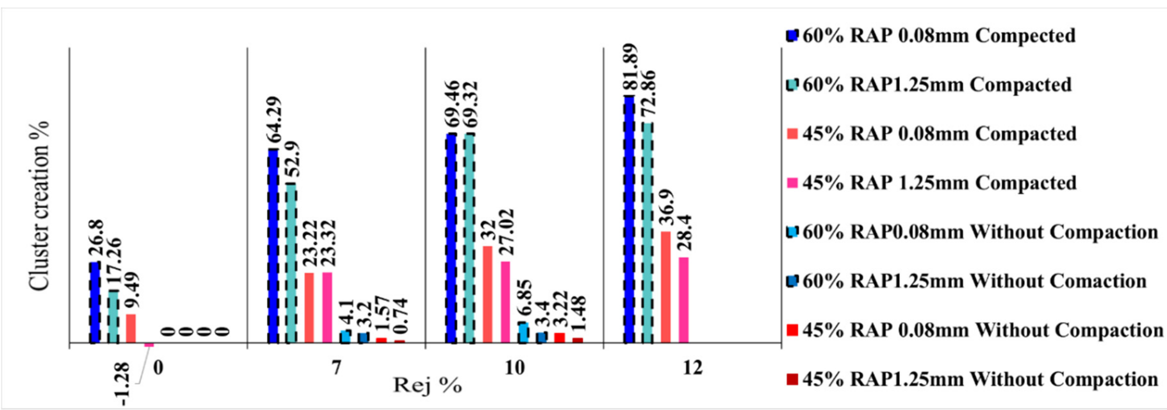


Figure 3.8 Effect of RAP content and size on the cluster formation

To continue the above discussion, Figure 3.9 present the effect of RAP content on clustering creation during the 6 weeks curing time without any additive. Results show that the specimens that have not been compacted (or in other words, RAP and aggregate were only mixed with spoon) had no cluster creation even after 6 weeks.

On the other hands, for the compacted specimens that had mechanical force to create cluster, specimens with 60% RAP content had about 3 times more cluster creation compared to the specimens with 45% RAP. It can be seen that the specimen with 60% RAP content and 0.08mm size, had 26.8 % cluster formation while the specimen with 45% RAP and same size, had only 9.5% increase for the first week. It should be noticed, the specimen with 60%RAP with even bigger size (1.25mm) had cluster creation of about two time more than the specimen with 45% RAP and 0.08mm size (17.26% and 9.49%, respectively). Therefore, it can be said that the amount of RAP has stronger effect and more important role on the cluster creation both for the specimens with and without additives. Besides, it can be seen that when the specimens had no rejuvenators and compaction, curing time had no effect on the cluster creation

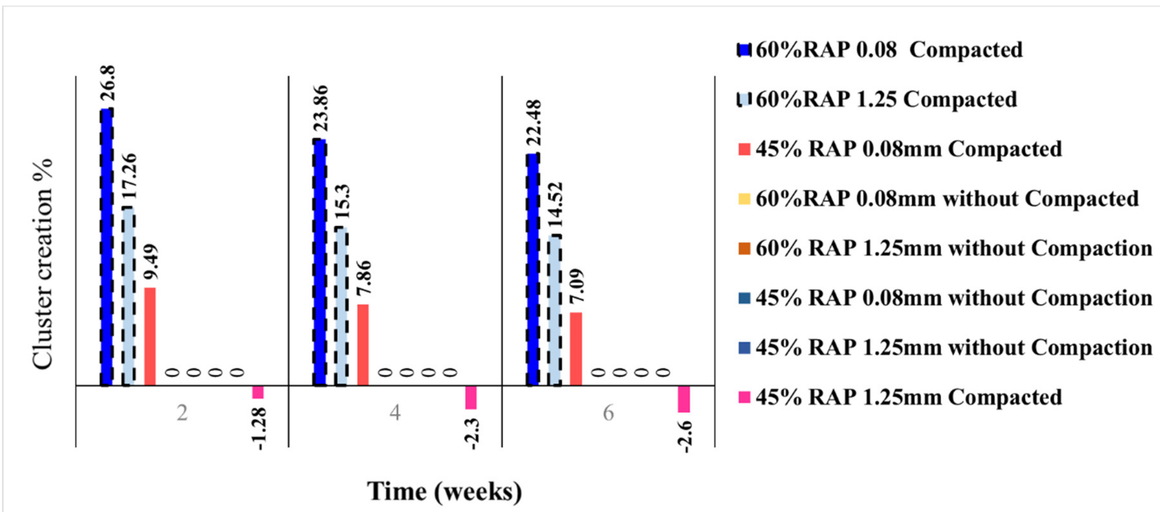


Figure 3.9 Cluster creation on the specimens without rejuvenator in different curing times

To compete this discussion, cluster creation of compacted RAP in different amounts and size including different amounts of rejuvenators (tracked during 6 weeks) is presented in Figure 3.10. In this figure, all blue family curves are related to 60% RAP content specimens and all red family are corresponding to 45% RAP content specimens. Besides, all columns with dot markers are related to RAP1.25mm and the other ones are those for RAP0.08mm.

Figure 3.10 also shows that when rejuvenator is added to the specimens, all specimens that contain 60% RAP have more amount of cluster creation compared to the specimens that contain 45% RAP. The highest amount of cluster is created by the specimen 60% RAP and 0.08mm size including 12% rejuvenator which is 81.89% in first 2 weeks. While, the lowest cluster creation is for the specimen 45% RAP and 1.25mm that contains zero percent rejuvenator. It can be seen that this trend could be tracked for all the specimens during the time when they have been examined.

In adition , Figure 3.10 shows the effect of curing time on cluster creation for 2 different amount of RAP (45% and 60%) and sizes (0.08mm and 1.25 mm). In both size of RAP(0.08mm,1.25mm), ratio of cluster creation is negative. As it can be seen, after 2 weeks (week 4th), amount of cluster has decreased slowly and this decreasing had continued in the next 2 weeks of curing time (week 6th) for all specimens. For example, for a specimen with 60% RAP in both size (0.08 and 1.25mm) the cluster formation in the second week was 26.8% and 17.26 % respectively, however, these values decreased in the subsequent measurements with longer curing times. This can be illustrated by the fact that sieving the specimens resulted in breaking the clusters. It should be noted that curing time might have positive effect on creating new clusters but to evaluate the amount new cluster creation new technic without mechanical force (sieving) can be suggested here for future works and studies.

Finally, Figure 3.10, also shows that, in a case where there is no additive in the mix and mechanical forces are existing, compaction might have negative effect during the time (refer to specimen 45%RAP.125mm). This is great importance as this is what is happening in the daily practice of asphalt pavements and their usage where the asphalt is experiencing daily loading during the time resulting in breakage of clusters.

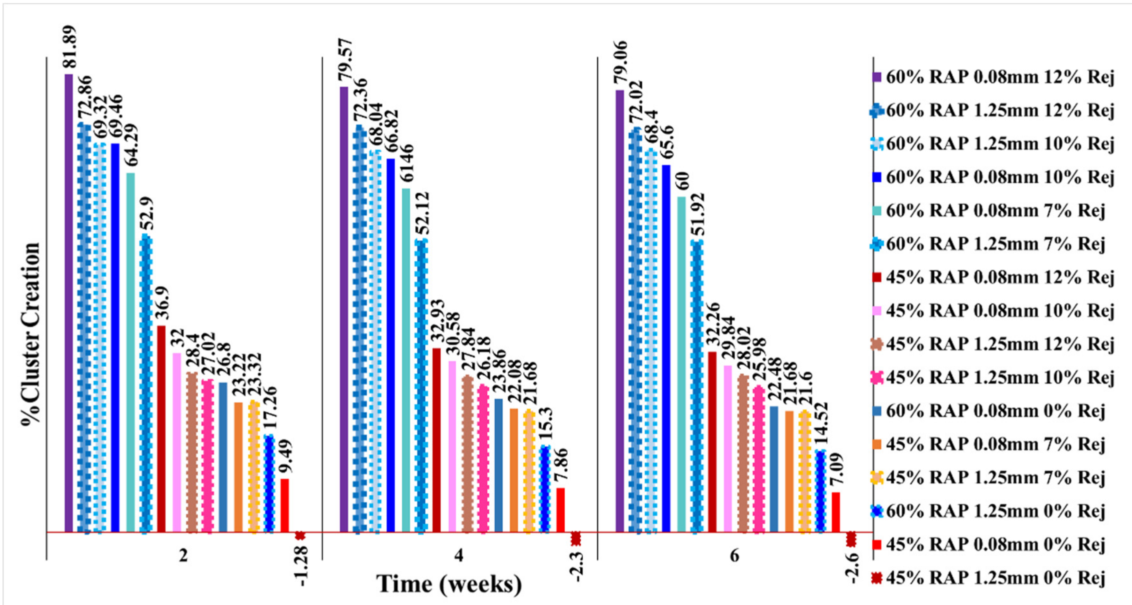


Figure 3.10 Graphical comparison of compacted specimens RAP content and RAP size effect on the cluster creation with different amounts of additive during the time

Figure 3.11 depicts the effect of curing time on cluster creation on RAP without compaction in the presence of different additive content. This graph illustrates that the curing time was more important for the specimens without compaction in comparison to the compacted specimens because here we see a different trend (even it is very weak and negligible). It can be said that the uncompacted specimens need more time to steak together. It can be seen that, the cluster creation for the uncompacted specimens decreased after 2 weeks but it slightly increased in the measurements of 4th week. This negligible effect is important for uncompacted specimens as the cluster creation rate here is very weak and these specimens on the contrary to the compacted specimens had not mechanical force for the cluster creation. Besides, it can be seen that adding 7% and 12% of additive could change the amount of cluster even without applying compaction, from 0% to 4.1% and 6.85% respectively.

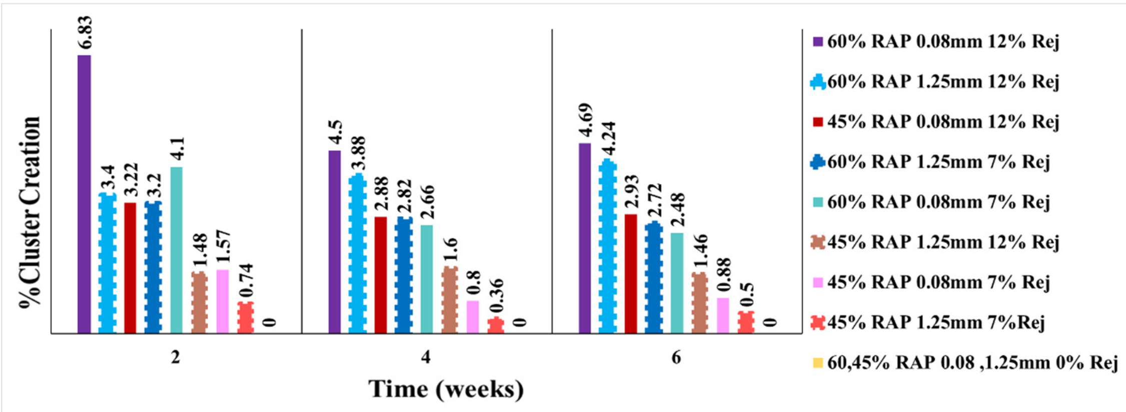


Figure 3.11 Effect of curing Time on cluster creation on without compaction RAP

Comparing Figure 3.10 and Figure 3.11 shows that when the compaction was added to the process the cluster creation was increased significantly. Table 3.1 shows the rate of cluster creation in the case of compaction and absence of compaction for four typical specimens of the present study with 12% rejuvenator. It can be seen that the usage of compaction could result in up to 21.42 times more cluster creation in the case of the specimen with 60%RAP and 1.25 mm size. A very important point in this table is that the effect of compaction in the specimens with coarser was more significant. As it can be seen, the compaction for the specimen (60% RAP) with the size of 1.25 had 21.42 times more cluster creation compared to the uncompacted specimen while this value for the size of 0.8mm was 12.59 times. The same trend is observed for the specimen with 45% and also other specimens with different amount of renovators. This could be because of the fact that the compaction in the coarser size, breaks the sizes to the lower sizes (fine particles) which in turn has more tendency to create clusters in the presence of rejuvenator compared to the coarse particles.

Table 3.1 Compacted vs uncompacted specimens cluster creation %

	60%RAP 0.08mm	60%RAP 1.25mm	45%RAP 0.08mm	45%RAP 1.25mm
Compacted	81.89	72.86	36.90	28.4
Uncompacted	6.54	3.4	3.22	1.48
Comp/UnC	12.59	21.42	11.45	19.18

Figure 3.12 is presented to visualize the average effect of RAP content and RAP size on the cluster formation in the whole compacted specimens. This figure shows the contribution of each group of specimens on the whole measured cluster creation in different specimens with different amounts of rejuvenator. To calculate the number in this graph first the summation of cluster creation amount in all specimens (different RAP content, rejuvenator content, RAP size etc.) were calculated. Then the summation of cluster creation in specimens with 60% RAP (including two RAP sizes) or 45% RAP were divided by the total value to see the contribution of the specific parameter (RAP content % and RAP size mm) on the measured cluster creation.

This graph clearly shows that smaller size, 0.08mm, has higher numbers of cluster in each series (60%, 45%) compared to bigger size. On the other hand, the contribution of 60% RAP content on the whole measured cluster formation was about 72 % (38%+34%) (i.e summation of 60%RAP0.08mm+60%RAP1.25mm) while this value is about 28% (16%+12%) for 45% RAP content. It shows the overall contribution of 60 % RAP content is 2.57 times higher than 45% RAP content. The same argument applies for RAP size, where RAP0.08mm contributed to 54% (summation of 38% and 16%) of total measured cluster creation while, RAP1.25mm contributed to 46% (34%+12%) of cluster creation. Therefore, RAP0.08mm had 1.17 times higher contribution to the cluster creation (see following average calculation).

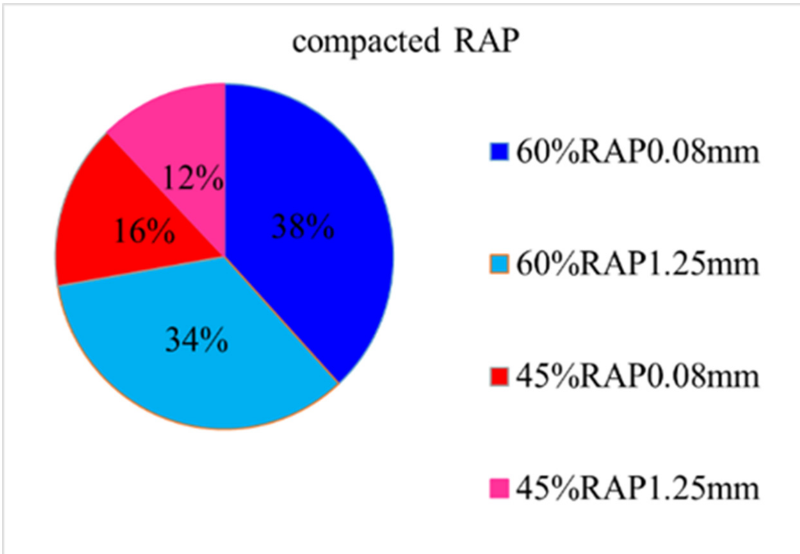


Figure 3.12 Statistically driven average correlation of RAP content and size alteration effect on the cluster formation in this setup of experimented materials.

$$RAP\ Content\ contribution\ on\ cluster\ creation = \frac{cluster\ 60\%}{cluster\ 45\%} = \frac{38+34}{16+12} = \frac{72}{28} = 2.57$$

$$Grade\ size\ contribution\ on\ cluster\ creation = \frac{cluster\ RAP0.08mm}{cluster\ RAP1.25mm} = \frac{38+16}{34+12} = \frac{54}{46} = 1.17$$

To compare the effect of strength of compaction and rejuvenator addition in the cluster creation, results of 4 series of specimens are provided in the table 3.2. It can be seen that the pure compaction had significant and more effect compared to the highest percentage of rejuvenator additions which in turn shows the importance of compaction in this process. It should be noted that the combination of compaction and additive to mix significantly increased the cluster creation (more than three times of superposition of these two modifications) showing the importance of this mixed process usage in the specimen preparation of cold mixes.

Table 3.2 Compaction vs Rejuvenator content effect on the cluster creation

RAP / Rejuvenator	60%RAP 40%Agg (Cluster creation %)		45%RAP 40%Agg (Cluster creation %)	
	0.08mm	1.25mm	0.08mm	1.25mm
0% Rej (only RAP+Agg)	0%	0%	0%	0%
0 % Rej on (RAP+Agg) + (compaction)	26.8 %	17.26 %	9.49 %	-1.28 %
12 % Rej on (RAP+Agg) + (rejuvenator)	6.8 %	3.4 %	3.2 %	1.48 %
12 % Rej on (RAP+Agg)+ (compaction + Rejuvenator)	81.89 %	72.86 %	36.90 %	28.4 %
	Synergistic effect (amount of cluster creation of specimens that has all variables is more than the sum of the cluster creation of each of them separately).			

The following conclusions can be derived from the discussions in this section:

1. Increasing in the amount of rejuvenators will increase the cluster creation. In this setup of materials cluster creation this is the observed trend:

   Cluster creation in specimens with 12%rejuvenator> Specimens with10%rejuvenator> Specimens 7%rejuvenator > Specimens 0%rejuvenator

2. In the case of RAP content and RAP size in each series of specimens, amount of cluster creation can be tracked as follows:

60%RAP0.08mm>60%RAP1.25mm >45%RAP0.08mm >45%RAP1.25mm

3. Order of Specimen preparation variables effect on the cluster creation:
   Compaction + Rejuvenator addition > Compaction > Rejuvenator addition > Amount of RAP in the mix> Size of RAP> Curing time

3.3 IDT

The indirect tensile (IDT) strength tests were carried out to characterize the resistance of mix asphalt. To do so, after preparing all the 24 specimens (as explained in section 2.1.3) and keeping them according to the curing time (two weeks at room temperature), the specimens were kept in water tank in room temperature for 2 hours before starting tests. The water bath and IDT test device are shown in Figure 3.13.

(a) (b)

Figure 3.13 A view of (a) conditioning of the specimens in the water tank; (b) IDT test on conditioned specimen

3.4 Effect of Rejuvenator Content and Preparation Temperature on the Tensil Strength

The first examined variable on the IDT test is preparation temperature. To this end, two different temperatures, 60℃ and 40℃, were selected. Two specimens with no added rejuvenator as the baseline were chosen to understand the effect of temperature without the intervention of rejuvenator. The IDT test results were interpreted comparing the strength of the specimens as a result of deformation during the test (Refer to section 2.1.3 for details of the IDT test). Figure 3.14 shows the results at 40℃ and 60℃. As can be seen at the same deformation level, the specimen prepared at 60℃ had almost 2.5 higher strength compared with that at 40℃. This implies that by increasing the mix temperature as much as 20 degrees, the resulting strength was affected considerably which emphasizes the importance of the effect of the specimen preparation temperature on the cold mix mechanical performance.

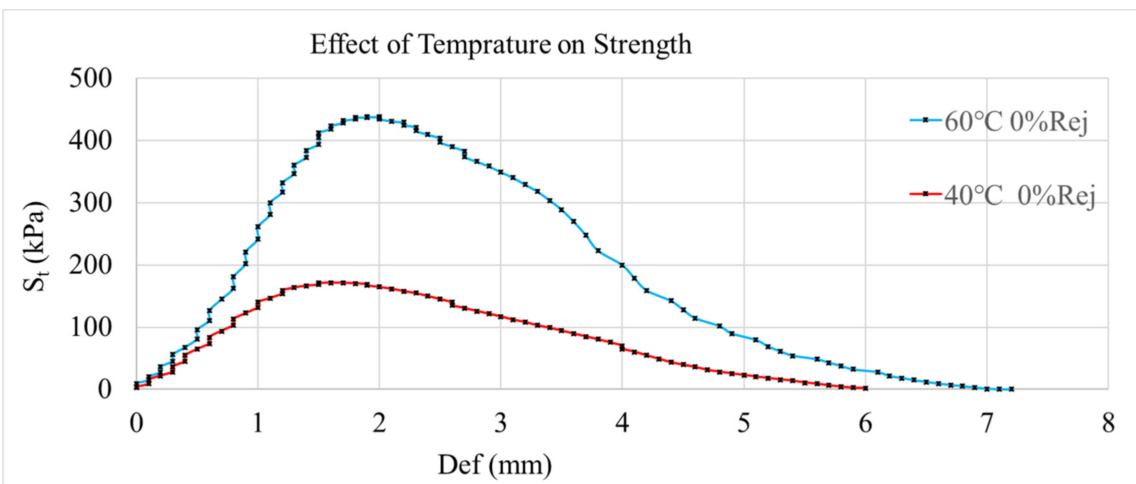


Figure 3.14. Effect of preparation temperature on resulting average strength of the cold mix with zero rejuvenator

$$S_t = \frac{2000*P}{\pi*t*D} \tag{3.1}$$

Where:

S_t = IDT Strength, kPa

P = Maximum load, N

t = Specimen height, immediately before test, mm and

D = Specimen diameter, mm

The second studied variable was the effect of the rejuvenator on the performance of the cold mix. Figure 3.15 (a) illustrates the effect of the rejuvenator dosage on the indirect tensile strength of specimens fabricated at 60°C. It can be recognized that, the lowest strength was pertinent to the specimen without any rejuvenator, which was prepared with 100% RAP. However, by adding the rejuvenator to the RAP, the strength values were improved. For instance, adding 2% rejuvenator to the RAP could enhance the strength from 440 kPa (i.e. zero rejuvenator) to 560kPa. Further addition of the rejuvenator into the mix by two more percent (i.e., 4% totally) resulted in the maximum strength of 680kPa. Nevertheless, the higher rejuvenator content (7%, 10%, 12%) led to a reduction in indirect tensile strength. The similar trend for the specimens prepared at 40°C, as shown in Figure 3.15 b, was observed in which the lowest strength (175kPa) was associated to the specimen without any rejuvenator (in accordance to what has been detected in previous series on 60°C). In the same way, by adding 2% rejuvenator the strength was almost doubled (i.e., 300 kPa). However, at 40 °C mix temperature, the highest strength occurs to the specimen containing 7% rejuvenator. It can be implied that the reduction in the mix temperature (i.e., from 60 to 40 °C) resulted in higher demand in the rejuvenator dosage in order to obtain the highest tensile strength at that specific temperature.

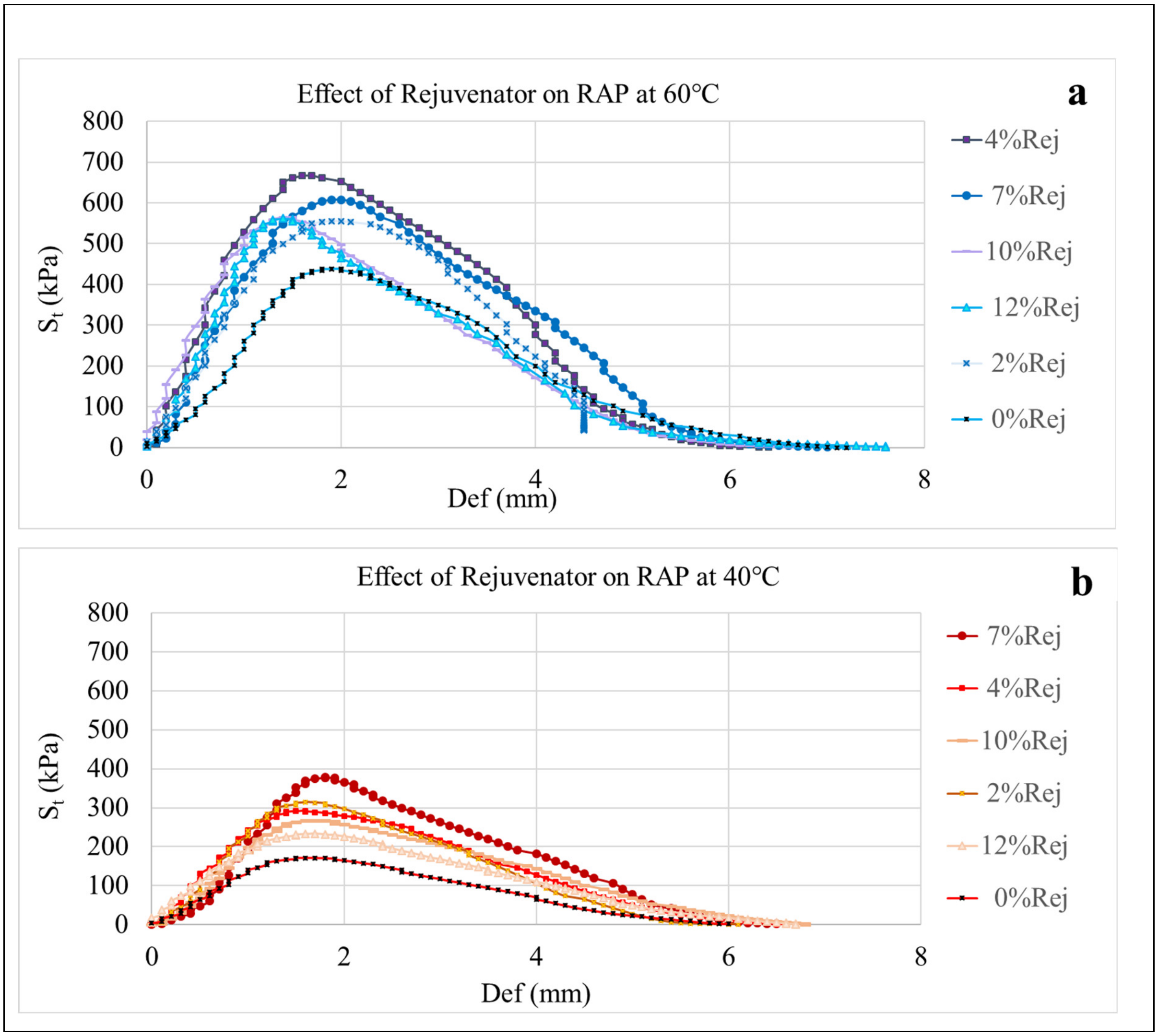


Figure 3.15. The indirect tensile strength of the RAP at different dosages of the rejuvenator at (a) 60 °C; (b) 40 °C

To have a better understanding of the results of IDT test on the RAP with zero and optimum rejuvenator content at two different temperatures (i.e., 40°C and 60°C), a comparative display is presented in Figure 3.16. It is evident that, at 60°C, the indirect tensile strength of the specimens with optimum and without rejuvenators, are considerably higher than the corresponding specimens at 40°C. Also, it can be seen that lowering the mix temperature by 20°C resulted in losing almost half of the strength (for example at optimum rejuvenator content, from 667.48 kPa for specimens prepared at 60°C to 377.77 kPa for specimens prepared at 40°C).

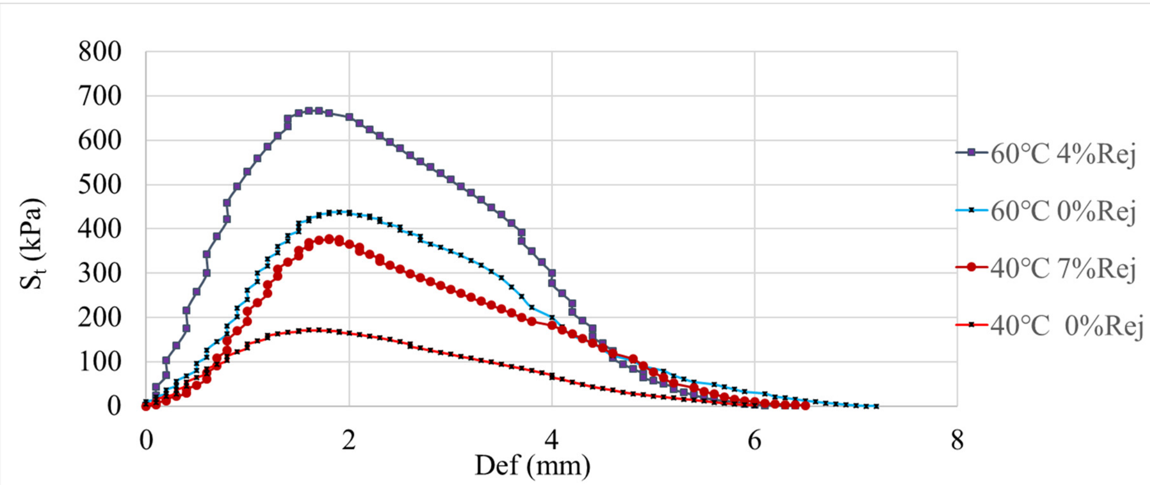


Figure 3.16 A comparison of the indirect tensile strength obtained in zero and optimum rejuvenator contents at 60 °C and 40 °C

Finally, Figure 3.17 demonstrates a comparison of all the results, discussed earlier, in order to find out the importance of two variables, studied in this section, on the mechanical performance of the RAP subjected to the IDT test. As shown, all blue lines represent the specimen that have been manufactured at 60°C and all red ones correspond to those made at 40°C. It can be realized that specimens that have been prepared at 60°C (regardless of the rejuvenator content) ehave higher strengths compared to the specimens prepared at 40°C. However, the change in the rejuvenator dosage only caused a slight improvement in the strength of the resulting modified material. According to the results and discussion provided, the impact of the mix temperature can be regarded as more influential factor on the IDT results.

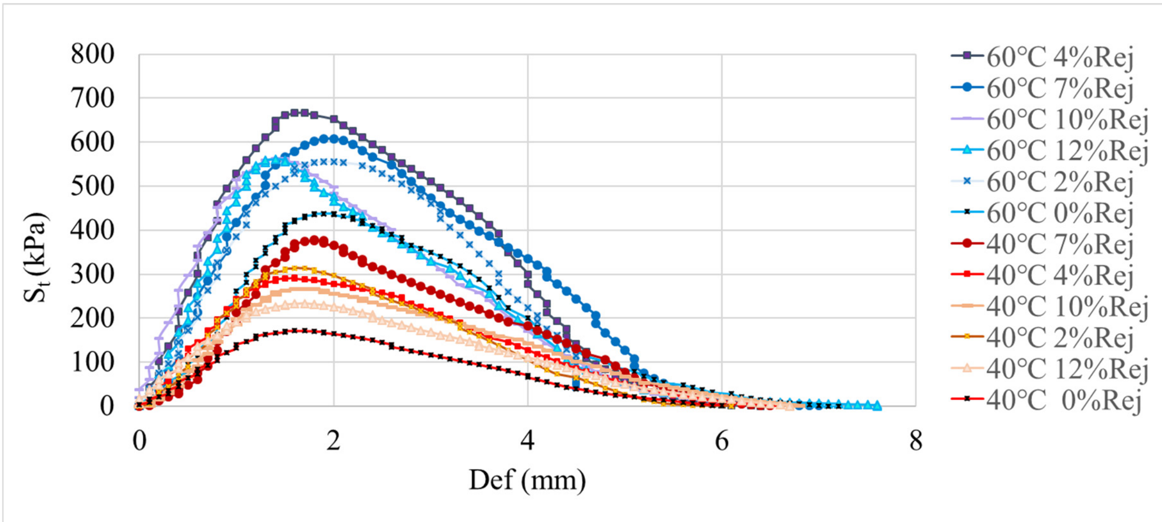


Figure 3.17 A comparison of the results of the IDT test on the RAP in different mix temperatures and the rejuvenator dosages

3.4.1 Energy Level

In the Figure 3.19, the bar chart compares the level of energy, dissipated during the IDT test, in different quantities of rejuvenators (0,2 %, 4%, 7%, 10%, 12%) that have been mixed with RAP at 60°C and 40°C in order to evaluate the effect of the rejuvenator content and mixing temperature on the energy level. The level of energy was calculated using integral of each curve (see in Figure 3.18) shown in strength/deformation analysis of Figure 3.17.

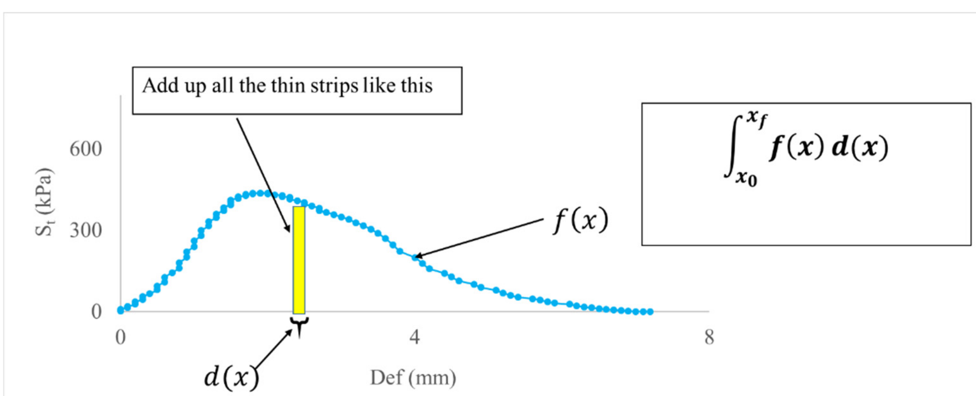


Figure 3.18 Typical calculation of energy using integral calculation of strain/ deformation curve

Figure 3.19 shows that, in total, the energy level of all the specimens prepared at 60℃ is more significant than those of specimens made at 40℃, regardless of the quantity of the rejuvenator used in the mix. Moreover, it can be seen that even the minimum energy level calculated for the specimens without the rejuvenator and mixed at 60℃ (i.e., 1374.79 J) was remarkably higher than the maximum energy level computed at 40℃ associated to the specimen with 7% rejuvenator (i.e., 1092.46J). Therefore, regardless of the rejuvenator content, specimens mixed at higher temperature had more energy, required before the failure during the IDT test, which could be translated as the importance of the mix temperature on the mechanical performance of the specimens prepared with different rejuvenator dosages. In other words, increasing the mix temperature helps to have a better resistance to failure in the IDT test as compared to the rejuvenator content.

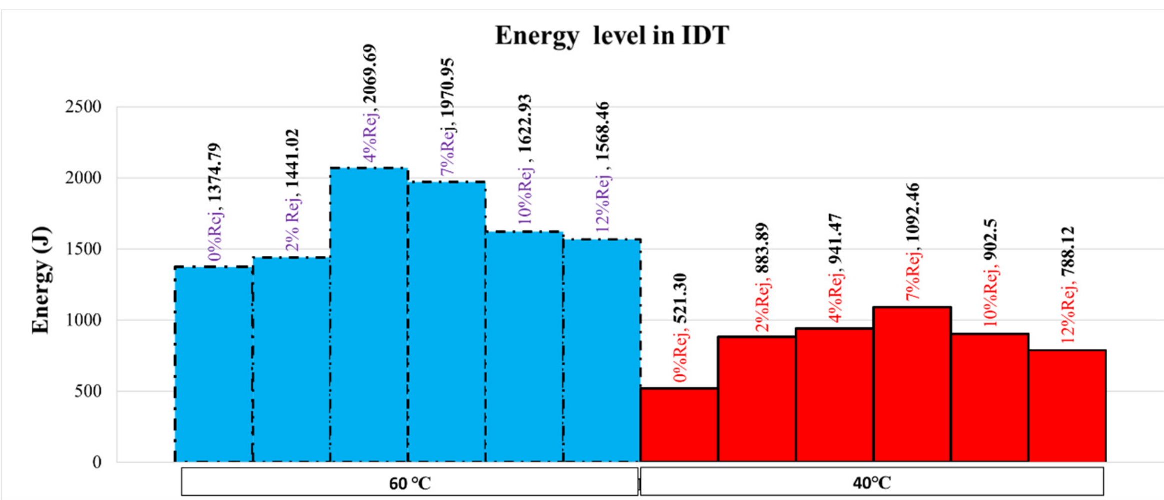


Figure 3.19. A comparison of energy level calculated in different mix temperatures and rejuvenator dosages

Moreover, it should be noted that the energy levels of specimens with more than 7% and less than 4% of rejuvenator were gradually decreased. This means that there is a specific amount of rejuvenator content in the mix for having an optimum (maximum) strength/energy level of the specimens according to their preparation and testing situation (like temperature, RAP bitumen content, etc.). It is clear that adding extra rejuvenator (more than optimum) to the specimens had negative effect on the level of energy and strength. In this setup of materials and tests, a similar trend of increasing and decreasing of energy levels can be tracked for both groups of specimens. For the group of specimens prepared at 60℃, the highest level of energy is related to the specimen with 4% of rejuvenator, while 7% of rejuvenator content resulted in highest energy among specimens prepared at 40℃. Therefore, it can be concluded that the optimum value of rejuvenator content for maximum energy level was increased by decreasing the preparation temperature. In other words, preparing cold mix material at lower temperature necessities a higher amount of rejuvenator to keep the design strength. Given the numbers available numbers of optimum rejuvenator content and their corresponding preparation temperatures (4% at 60℃ and 7% at 40℃) it can be said that digressing 10 degrees in specimen preparation necessities increasing 1.5 % rejuvenator addition to the mix to have the optimum strength at that specific temperature.

3.5 Effect of Rejuvenator Content on Extracted Binder behavior

In order to shed light on the effect of the rejuvenator on the mechanical properties of the bitumen, already used in the RAP, different dosages of the rejuvenator were applied to the specimens made of the RAP. After the compaction (by SGC), they were left for 2 weeks at room temperature to be completely cured and then the bitumen was extracted from the specimens. In each rejuvenator content, in order to obtain enough material, two to three times of the extraction were carried out (4 kinds of bitumen were extracted from: 100% RAP, RAP+4%Rej, RAP+7%Rej, RAP+12%Rej). At the end, four sets of tests were employed which will be explained in detail in the following subsections.

3.5.1 Low temperature (BBR)

The aim of this test is to measure the low temperature stiffness and relaxation of binder over the time to determine the resistant of binder to low temperature cracking (details of BBR test are provided in section 3.1.3.1).

3.5.1.1 Stiffness

The BBR test is employed to determine the lowest temperature that asphalt binder can undergo before cracking (stiffness as a function of time). Based on the conclusions driven from the IDT test, it is assumed that by adding the rejuvenator into the mix, bitumen properties would improve.

Two factors were evaluated in this test, as explained in the standard procedure:

- Creep stiffness (S) is the lowest temperature that binder can resist before first crack under 300 MPa pressure occurs. It should be noted that exceeding the stiffness higher than 300 MPa increases the probability of the appearance of cracking, therefore 300 MPa is normally selected as the maximum limit.
- M-Value refers to the bitumen hardening slope which is determined from master stiffness curve slope (stress relaxation parameter). By increasing the m-value, the probability of the occurrence of thermal cracking increases. Hence, 0.3 is determined for the minimum limit for this value.

$$Sm(t)= \frac{P\ L3}{4\ b\ h^3\ \delta(t)} \qquad (3.2)$$

Sm(t) =	Creep stiffness (MPa)
P =	Test load (N)
L =	Distance between supports (mm)
b =	Width of specimen (mm)
h =	Thickness of specimen (mm)
δ (t) =	Deflection at time t (mm)

LT_s = Low temperature (driven from stiffness curve)

LT_{m-v} = Low temperature (driven from m-v curve)

LT= Minimum size number between LT_s and LT_{m-v}

LT_e = Low temperature effective (resistant against first crack)

Figure 3.20 shows the evolution of creep stiffness of specimens prepared with different rejuvenator content over a range of temperatures with reference to the maximum value of pressure equal to 300Mpa. It can be seen that by adding 4% rejuvenator to the RAP, low temperature performance (LT_s) of bitumen was increased by one size (reduced by 6 °C) compared to the specimen without rejuvenator. The same improvement of one size can be observed in comparing the results of specimens with 4% and 7% of rejuvenator content. Furthermore, by adding 12% of rejuvenator, LT_s was improved from -26 °C to -46 °C which means a meaningful increment in low temperature performance compared to the virgin. It is worth mentioning that -26 is not a PG size that exists in the market and it is only driven from crossing with the reference line. Therefore, maximum amount of rejuvenator content resulted in maximum improvement in the low temperature creep stiffness. In other words, an increase in the percentage of rejuvenator in bitumen led to an increase in tolerance of bitumen against the manifestation of the first crack in lower temperature condition.

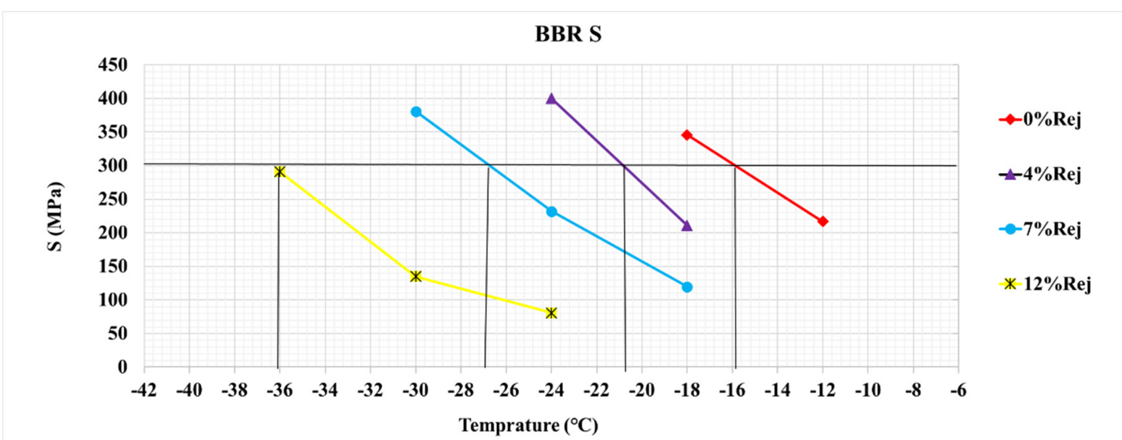


Figure 3.20. Evolution of creep stiffness over temperature in different rejuvenator content used in RAP

Figure 3.21 shows the effect of the rejuvenator content on the m-value of extracted bitumen. Similar to low temperature creep stiffness curve, by increasing the rejuvenator content, the mechanical performance of bitumen in lower temperature was improved. As can be seen, bitumen without rejuvenator at -12.6°C had the same stress relaxation level as the one with 12% rejuvenator at -26.5°C. In other words, adding 12% rejuvenator could contribute to maintaining the performance of bitumen even after decreasing the temperature by about 14°C. Similarly, $LT_{m\text{-}v}$ changed from -12.6 to -19.4°C by adding 4% rejuvenator which means 6.8°C temperature improvements. By adding 3% more of additive (7% totally) $LT_{m\text{-}v}$ improved to -24°C which means further 4.6°C improvement after the second change. However, adding another 5% additive (in total 12% rejuvenator) could only contribute to 2.5°C improvements (i.e., -26.5 vs -24 in the specimen with 7% rejuvenator content). This shows that the improvement in the low temperature performance of the bitumen in the presence of the rejuvenator is limited at higher contents.

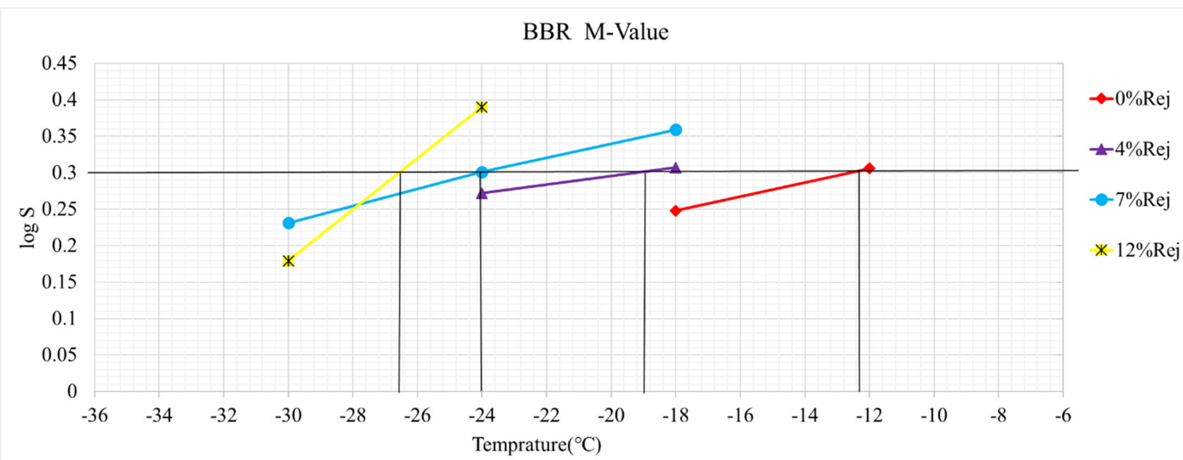


Figure 3.21. Evolution of m-value in different rejuvenator content used in RAP

Table 3.3 provides a summary of the results discussed in Figure 3.20 and Figure 3.21. The LT value (maximum of $LT_{m\text{-}v}$ and LT_{s}) is also driven and presented in this table. It can be seen that the LT of specimens without rejuvenator was improved from -24 to -34 by adding 7% of additive. It is worth mentioning that, although the specimen with 12% rejuvenator had a LT_s of -34°C, it turns out the same LT as in the specimen with 7% (-34°C).

Table 3.3. A summary of low temperatures obtained from the creep stiffness and m-value

Rejuvenator%	LT_s	LT_{m-v}	LT
0	-22°C (-12 + -10)	-22°C (-12 + -10)	-22°C
4	-28°C (-18 + -10)	-28°C (-18 + -10)	-28°C
7	-34°C (-24 + -10)	-34°C (-24 + -10)	-34°C
12	-46°C (-36 + -10)	-34°C (-24 + -10)	-34°C

3.5.1.2 ΔTc

ΔTc is an index to quantify the potential of ageing in bitumen that is obtained from subtracting the BBR creep or m-value temperature from the BBR stiffness temperature.

$$\Delta Tc = (LTs\text{-effective} - LTm\text{-effective}) \tag{3.3}$$

For instance, if LTs-effective (BBRs' temperature) is -17.3°C and LTm-effective (m-value's temperature) is -12.4 °C then ΔTc = (-17.3 - (-12.4)) = -4.9. It should be noted that binder size is m-value controlled.

Importance of ΔTc: The importance of the study of the ΔTc can be viewed from different perspectives:

- Increasing the binder m-contorted lead to increasing negative ΔTc;
- Increasing negative ΔTc leads to increasing the probability of the top-down fatigue cracking of pavement;

- Although it is not obvious in the first sight, the value obtained in the low temperature testing should be considered to be associated with distresses which in turn have correlation with intermediate service temperatures.
- ΔTc has a relationship with pavement surface distresses.
- ΔTc is related to non-load thermal cracking (contraction and expansion) and potential of aging;
- In the final analysis ΔTc comes down to binder relaxation.

Table 3.5 presents the calculations of ΔTc based on the results obtained from the BBR test. It is noticeable that the bitumen extracted from the RAP containing 4% rejuvenator had the lowest ΔTc among the tested materials. This means this the bitumen extracted from the specimen with 4% additive content had the maximum ability against in this set of materials.

Table 3.4 ΔTc calculation for different dosages of the rejuvenator in the RAP

Rejuvenator%	**$LT_{e\,S}$**	**$LT_{eM\text{-}V}$**	**ΔTc ($LT_{e\,S}$ - $LT_{eM\text{-}V}$)**
0	-15.9 °C	-12.5°C	-3.4
4	-20.9°C	-19°C	-1.9
7	-26.8°C	-24°C	2.8
12	-36°C	-26.5°C	9.5

3.5.2 High temperature

The results in Figure 3.22 show the results of dynamic shear rheometer (DSR) test on the extracted bitumen from specimens that contain different dosages of rejuvenator (0%, 4%, 7%, 12%).

There are two horizontal lines in this graph that show the limit of G*/sinδ (kPa) for aged and unaged bitumen.

- G*/sinδ =2.2 kPa for aged bitumen
- G*/sinδ =1 kPa for unaged bitumen

It should be emphasized that the rejuvenator cannot completely (100%) rehabilitate the aged bitumen, therefore these specimens considered as aged bitumen in this analysis and the G*/sinδ =2.2 kPa was used as the reference line. According to the results, at a specific temperature of 88°C, increasing the rejuvenator content in the extracted bitumen resulted in the reduction of G*/sinδ; the value of 2.4 was decreased to 0.9 by adding 7% rejuvenator. By considering the reverse correlation of G*/sinδ and dissipated work in each cycle of rutting, it can be concluded that the performance of bitumen was decreased by adding more rejuvenator content. Similarly, for a specific value of G*/sinδ (for instance G*/sinδ =2.2) it can be seen that an increase in the rejuvenator content in the extracted bitumen resulted in the reduction of the high-performance size of bitumen (from 89 °C to 80 °C by adding 7% percent of rejuvenator).

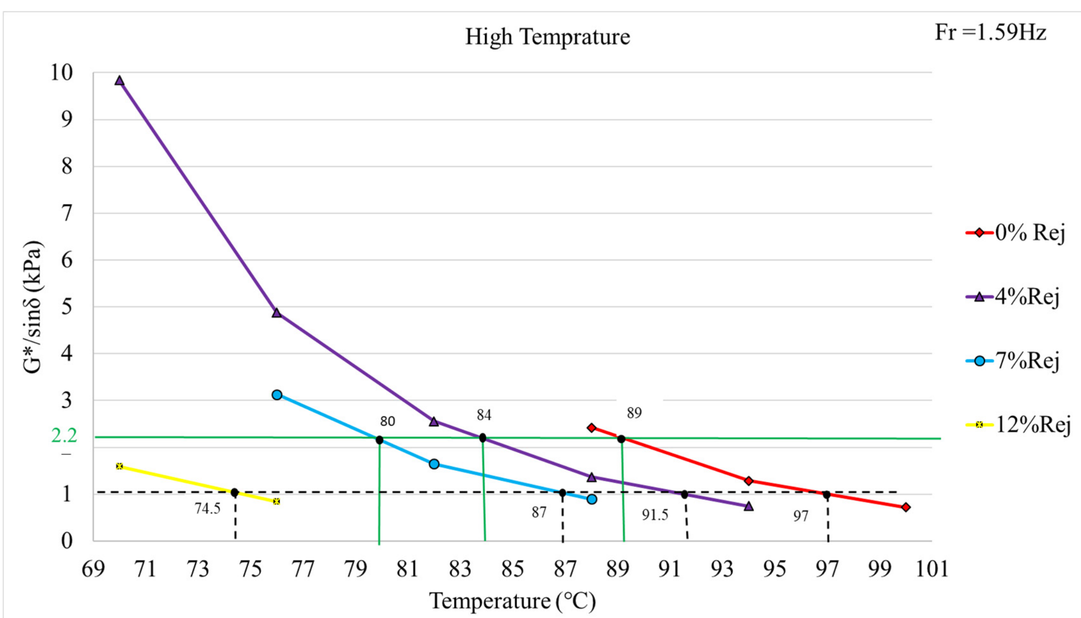


Figure 3.22. DSR test result on the bitumen extracted from the RAP mixed with different percentages of the rejuvenator.

This reduction of G*/sinδ was expected since that value increases when bitumen age and the rejuvenator is, as the name implies, suppose to rejuvenate, to reverse aging.

3.5.3 Shear complex modulus

Figure 3.23 shows the experimental results obtained from the shear complex modulus tests on old bitumen (extracted from 100%RAP) as well as the extracted bitumen from RAP combined with three different percentages of the rejuvenator (i.e. 4%, 7% and 12%). Figure 3.23a presents a comparison among all extracted bitumen in the form of the Cole-Cole diagram in which the storage shear modulus is plotted on the x-axis and the loss shear modulus is depicted on the y-axis. This presentation provides a suitable tool to assess the rheological behaviour of the bitumen at low and intermediate temperatures. However, the black diagram, as shown in Figure 3.23.b, is a more accurate way to understand the rheological behaviour of the bitumen at high temperatures. Both of these diagrams are used to calibrate a model (e.g., the 2S2P1D model) of the rheological behaviour of the bitumen. On this ground, it can be seen from the Cole-Cole and Black diagrams that all bitumen specimens, with different percentages of the rejuvenator, follows the time - temperature superposition principle as all the measured points fall onto a single curve. In other words, the rheological properties of the studied bitumen are predictable by using the 2S2P1D model.

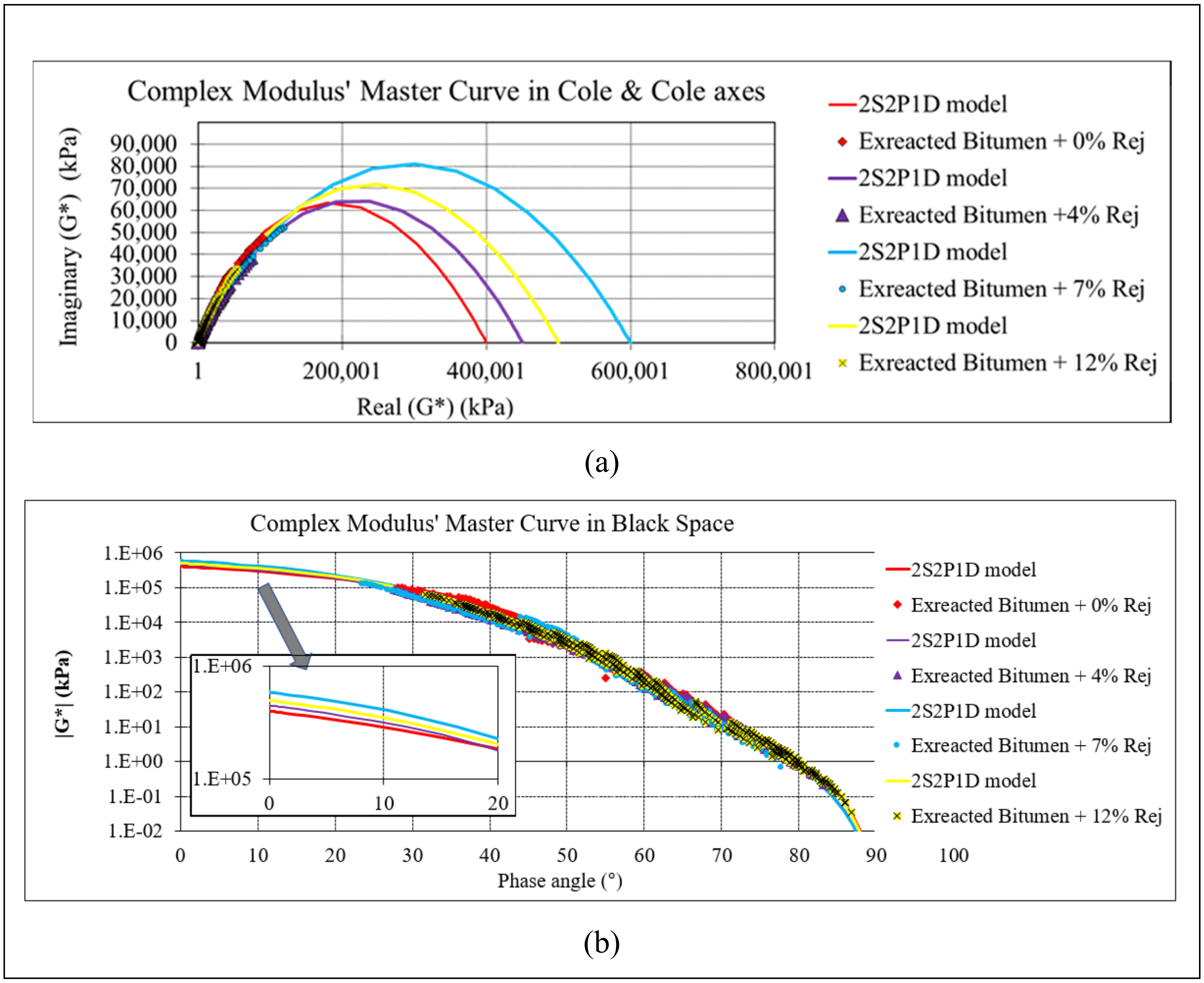
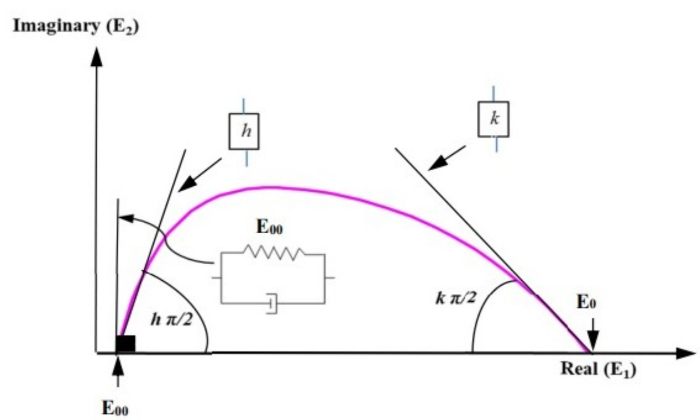


Figure 3.23. The DSR test results for the virgin and extracted bitumen at different percentages of the rejuvenator (a) Cole-Cole diagram (b) Black diagram

In this study the 2S2P1D (2 Spring, 2 Parabolic elements and 1 Dashpot) was used to describe the rheological behaviour of the virgin and modified bitumen because it more precisely describes the linear viscoelastic behaviour of the bitumen and its mix with aggregates in small strain field in any combinations of temperatures and frequencies. On this basis, Table 3.6 shows the parameters of this model obtained from minimizing the error between the experimental points and the points from the 2S2P1D (the solid curves) model. As can be seen, by adding more rejuvenator into the extracted bitumen, the glassy modulus (E_0) and the dimensionless constant (δ), used in the 2S2P1D model, were highly affected while the rest was left almost unchanged. It is worth mentioning that the highest amount of these two variables were obtained at 7% of using the rejuvenator in the extracted bitumen and the lowest one was pertinent to the bitumen without rejuvenator.

Table 3.5: A comparison of the 2S2P1D model parameters for extracted and virgin bitumen

Specimen (%of rejuvenator)	E_∞ (MPa)	E_0 (MPa)	k	h	δ	τ_E (s)	β	T_{ref} (°C)
Extracted (0%)	0	400000	0.350	0.620	2.5	0.00003500	580	34
Extracted (4%)	0	450000	0.345	0.645	5.6	0.00002200	380	34
Extracted (7%)	0	600000	0.330	0.648	7.0	0.00000430	450	34
Extracted (12%)	0	500000	0.340	0.630	4.5	0.00000035	500	34



The Time-Temperature Superposition Principle (TTSP) can be utilized to construct a unique curve in log |G*|-log.a_T.fr coordinates, as shown in Figure 3.24(a), at an optional reference temperature by applying a shift factor (a_T) to the |G*|-log.fr curve obtained at different temperatures. This presentation is called master curve. It provides not only a complete view into the stress-strain relationship at different frequencies and temperatures but also a way to predict the shear modulus of the bitumen beyond the range of tested frequencies. To this end, in this study the reference temperature was selected as 34°C and the master curve was constructed for the virgin and the extracted bitumen including different dosages of the rejuvenator. It was observed that the extracted bitumen including the rejuvenators showed higher variability than the extracted binder, for example figure 3.24 (b), which is complex modulus in a T_{ref}=34°C and different frequency, shows that for specimen extracted bitumen +4% rejuvenator this number is 3277 MPa while for specimen extracted bitumen without any rejuvenator is 6554 MPa. This variability becomes more significant as the dosage of the rejuvenator increases. Another finding is that, by adding more rejuvenator into the extracted bitumen, the shear dynamic modulus was negatively affected at low frequencies or high temperatures while this effect was not so prominent at high frequencies or low temperatures.

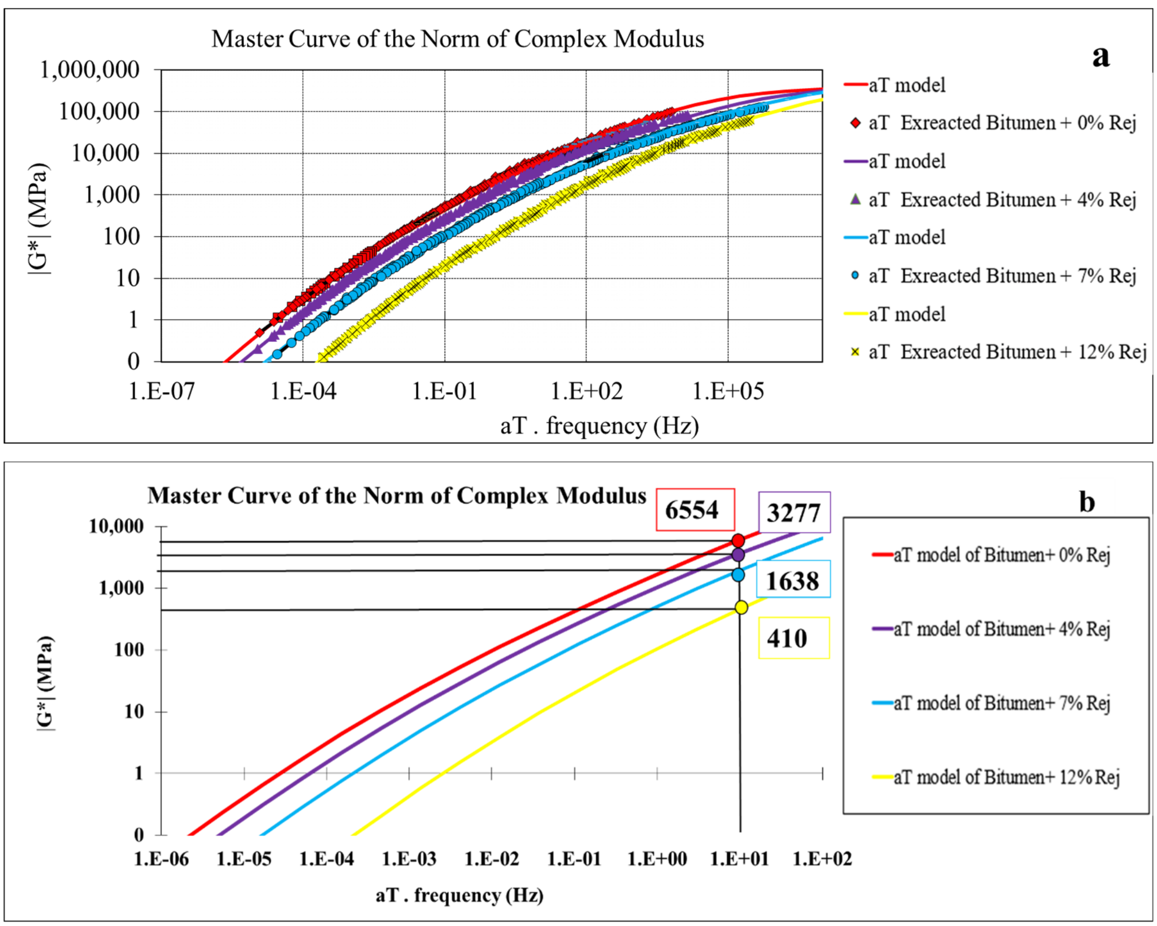


Figure 3.24 Master curve of the virgin and extracted bitumen at reference temperature of 34°C

As observed on Figure 3.24, the modulus decreases with the addition of rejuvenator. This demonstrates once again that the rejuvenator works, but it also means that higher strains would be observed in the field with the rejuvenated mixes compared to the standard CRM. This could result in the need to make thicker pavements to have pavement structure that last as long. Performance tests on the rejuvenated mixes are needed to better understand this part.

3.5.4 FTIR

As illustrated in chapter 2, FTIR (Fourier Transform Infrared Spectroscopy) test is a qualitative analysis used for identification of functional groups of materials. Also, this method can be used as a quantitative analysis when standard reference materials are available.

It should be mentioned that FTIR is a bulk analysis technique, this little information comes from small concentration of compound on a specimen (typically greater than 5% constituent). The FTIR analysis graphs include several peaks at different wavenumbers. Each peak at different wavenumbers has a specific identification to distinguish a specific component. The functional group region starts from 4000 cm^{-1} to 1450 cm^{-1}, and the fingerprint region includes the range between 1450 cm^{-1} to 500 cm^{-1}. The peaks of each component in the fingerprint region always occurs at the exact wavelength range with a unique pattern that can be distinguished from other components, same as a distinguishing fingerprints of people, which illustrates the reason behind this nomenclature. Although, in many FTIR results, specially for the complex components, the fingerprint range is too complicated, it can help to confirm the findings in the functional group region.

Figure 3.25 shows the FTIR spectra of 5 specimens that have different transmit peak with respect to analyzed range of wavelengths. These specimens include pure rejuvenator, asphalt new binder, old bitumen (extracted from 100% RAP), and bitumen extracted from specimens including RAP and different percentages of rejuvenator. As mentioned, in this test, the intensity of the absorbance/transmit is correlated to the quantity of functional groups in a specimen if the specimen perpetration process (like the thickness of specimens) and test conditions are kept equal. Although in this study it was not possible to keep an equal test condition for all specimens according to the quantitative analysis requirement, the peak intensity of specimens results presents an approximate amount of each functional group to justify the analysis. To analyze the results, the strong peaks will be discussed then the others will be tracked.

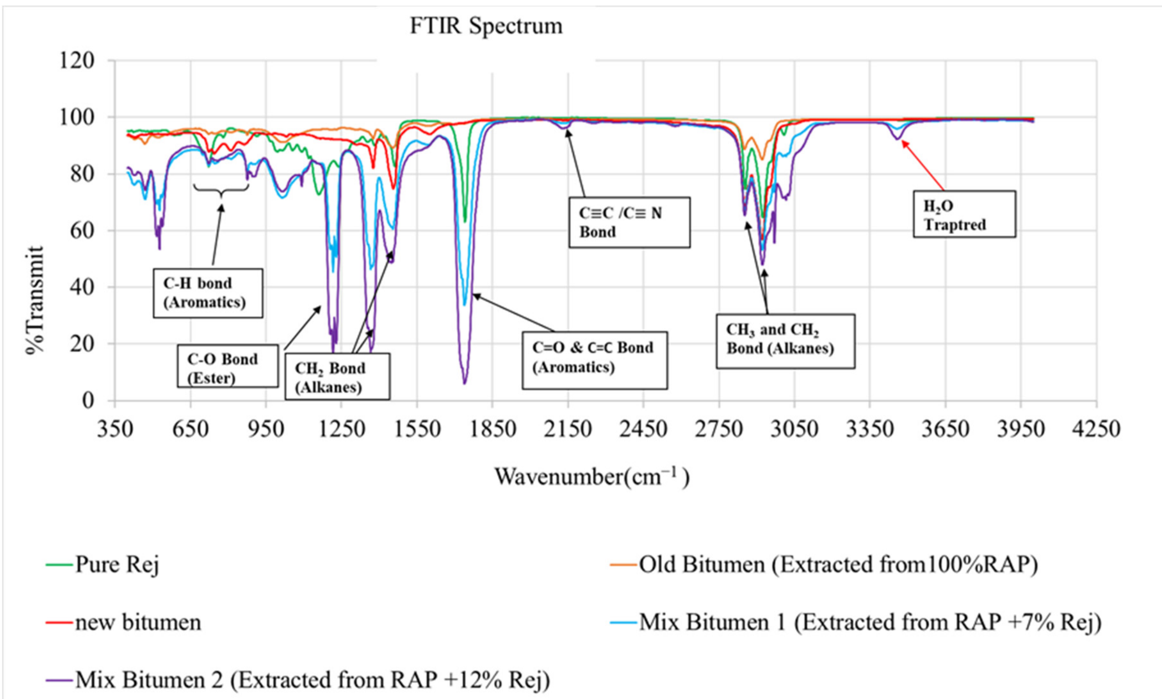


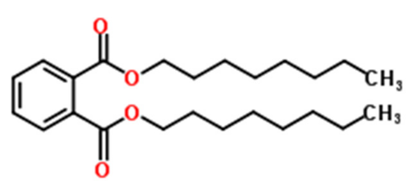
Figure 3.25 FTIR spectra results for different specimens including pure rejuvenator and different extracted and virgin bitumen.

The peaks at the range of 2900-3100 cm^{-1} represent the tensile bonds of alkane (CH_3) hydrocarbons. Considering the fact that according to the bitumen complex compound, the aliphatic (alkanes and alkenes) are important part of bitumen, having this peak in the FTIR analysis is consistent with the bitumen compound. It can be seen that, the intensity of these peaks (as basic part of organic material) in the specimen including pure rejuvenator (bio-binder), new bitumen and mix specimens (specimens with RAP + rejuvenator) are significantly more than the old bitumen (extracted from 100% RAP). This can be correlated to the fact that the old bitumen has been aged/oxidized by time (ageing of bitumen is because of oxidation of materials). In other words, during the oxidation process, the material compounds losing an electron/H^-, transform to the other structure like unsaturated compounds (C-CàC=C/C≡C) resulting in reduction of alkane quantity in the specimen. As it shows, the peak intensity of maximums (specimens with RAP + rejuvenator) is more than new bitumen, pure rejuvenator and old extracted bitumen. It is because of the synergistic effect that happens when oil /rejuvenator is added to the extracted bitumen and the performance of mixed specimens is more than the sum of the performance of each of them separately. The same behaviour was observed in mechanical testing results where the effect of compaction, rejuvenator, and curing time, altogether was much more e than summation of these individual effects together.

On the other hand, the next strong peak at 1750 cm^{-1} is related to a component with C=O group. As this peak appeared only in the specimens of rejuvenator and those that contain rejuvenator, it can be concluded that the identified C=O bond is part of rejuvenator structure. The same trend can be seen for the strong peak at 1050-1250 cm^{-1} which is the indicator C-O bonding in the component of rejuvenator and mixed specimens. Considering the fact that C=O and C-O important functional groups (including Aldehyde, Ketone, Acid,..) that can make several chemical reactions, we should investigate the rest of the graphs to see whether they could result/contribute in a specific chemical functional group or not. To do this, other peak differences between specimens should be examined. Beside as it shows, intensity of peaks in 1750 cm^{-1} follows this order: mix 2> mix 1> pure Rej. This peak conforms pseudo interaction that led to intermediate complex that can be separated with small energy and they are not persistent compound.

Firstly, coming to the weak peaks (usually these weak peaks are not that much important in FTIR spectroscopy) in the FTIR results the peaks at around 2150 $cm^{-1\ can}$ represent alkyne group (C≡C) or cyanide (C≡N). Since this peak appeared only in mix specimens, it can be said that this component is not part of the rejuvenator nor pure bitumen. It should be noted that to have cyanide (C≡N), Azote (N≡N) must be taken from air then broken to the N atoms and consequently start to react with carbon. Such a process needs significant reaction situation that requires a reaction energy equal to lightning, to break the triple bonds of nitrogen gas. Hence, considering the test situation of current study this peak cannot be cyanide and is related to the alkyne group, C≡C, which can be easily created during the oxidation. Therefore, no specific indication of chemical reaction between rejuvenator and RAP materials can be concluded here. Secondly, there is not any OH functional group (either alcoholic or acidic) in the mix materials as we cannot see a wide big peak around 3000 cm^{-1}-3600 cm^{-1}. Besides, the peak at 3350 cm^{-1} can be either an indicator of water (that can be created during any reaction or trapped from air) or the indicator of presence of unstable H band in components (that cannot transfer any important information about any chemical reaction). Hence, here also, no indication of chemical reactions can be correlated to previously identified C=O / C-O groups.

Therefore, in this setup of materials, it can be concluded that the rejuvenator's C=O/C-O bonds are not willing to react with other component of bitumen and they just remained intact in the extracted bitumen. This behaviour is close to the Ester group (RCOOR) that normally used as non-active solvent (e.g., Dioctyl Phthalate) in the mixes. The combination of C=O and C-O group presence (in parallel to the previously discussed aromatic and aliphatic structures) in the results can confirm the structure of the ester group in the rejuvenator. The structure of Dioctyl Phthalate including C=O, C-O, aromatic and aliphatic structures is shown in Figure26. It should be mentioned that the accurate estimation of the exact structure in the necessities a comprehensive NMR and GCMS testing. Although we were able to throw down the existence of chemical reactions using the FTIR test further chemical studies should be investigated to confirm the findings that can be suggested for future study in this field.



(Dioctyl Phthalate)

Figure 3.26 Structure of Dioctyl Phthalate

According to the FTIR results, the bitumen from the RAP is not modified chemically by the rejuvenator. However, the different rheological tests have shown the impact of the rejuvenator. From that, we can conclude that the rejuvenator, which is of low viscosity, simply dilute the RAP bitumen and the resulting mix could be considered as an average between the old RAP bitumen and the rejuvenator oil.

CONCLUSION

The main objective of the present study was to investigate the rejuvenator addition effect on the cold recycled materials properties. The specific objectives were to assess the physical and chemical interaction between the RAP and its bitumen content with the added rejuvenator. Experiment tests and plans were designed and conducted to analyze the rejuvenator effect in different aspects such as cluster creation in the cold recycled mix, RAP strength and rehabilitated bitumen's low temperature and high temperature performance. Besides, the chemical or physical nature of the rejuvenator effect on the bitumen content of the RAP was studied. From the results, it can be concluded that the addition of rejuvenator does have an impact on the CRM properties even if the rejuvenator used was designed to be used with hot mix asphalt. The following concluding remarks are drawn:

- Increasing the amount of rejuvenators will increase the cluster creation in the mix.
- In the case of RAP content and RAP size it can be said that the RAP content had more significant effect on the cluster creation where the cluster formation for the mix with 60% RAP in both size of 0.08mm and 1.25mm were higher than those of the mixes with 45% RAP content. It should be noted that the smaller size resulted in more cluster formation in each corresponding RAP content.
- Results indicated that the rejuvenator addition in combination with the specimen preparation protocol (such as compaction) could significantly increase the cluster formation. The following trend (highest cluster formation to minimum cluster formation) was found for the cluster creating with different scenarios of mix preparation protocols:

  Compaction + Rejuvenator addition > Compaction > Rejuvenator addition > Amount of RAP in the mix> Size of RAP> Curing time
- IDT test revealed that the RAP specimen preparation temperature had more effect compared to the rejuvenator addition where all the specimens prepared at 60°C had higher strength compared to the specimens prepared at 40°C. However, at each specific temperature an optimum value of rejuvenator content could be achieved which in turn reduced by increasing the specimen preparation temperature. In this setup of materials, 4%

of rejuvenator content was identified as the optimum additive content for the specimens prepared at 60℃, while 7% rejuvenator was identified as the optimum value for the specimens prepared at 40℃.

- It was concluded that an increase in the rejuvenator content of the RAP could significantly increase the low temperature and high temperature of rehabilitated bitumen (extracted from specimens with additives), where the maximum performance was observed for the specimen with maximum amount of rejuvenator (12% in this study). However, results indicated that the specimen with 4% rejuvenator could show the maximum ability against aging among all the examined rejuvenator contents.
- Finally, a cross-functional comparison of FTIR test results of virgin bitumen, rejuvenator and rehabilitated bitumen could not reveal any modification/production in the chemical functional group of rehabilitated bitumen. This suggests that the investigated commercial rejuvenator physically affects the RAP and mix by alteration physical properties such as viscosity.

RECOMMENDATIONS

After finishing the current study following extended investigations might be suggested to continue the research in this field.

- The first and foremost suggestion to continue the work would be doing NMR and GCMS tests to verify the conclusions on the chemical reactions between rejuvenator and bitumen in the recycled cold mixes. Conducting Rutting test on the modified mixes could demonstrate valuable results on the effect of rejuvenator on the cold recycled pavements.
- Investigation of temperature effect on the cluster formation could also be another important suggestion in this field to have more information on the specimen preparation protocols for the cluster creation.
- It is suggested to have another method of cluster creation in parallel to the sieving method to verify the results (because sieving breaks some of the clusters and diversely affect accuracy of the drawn values).
- Concerning the optimum rejuvenator content value identifications, it is suggested to redo the experiments in smaller intervals of rejuvenator percentage (more specimens) with more preparation temperatures to drive a general correlation in this regard

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