Performance Evaluation of Stabilized Base Courses Comprising TSRU Tailings and Bitumen Froth Derived from Alberta Oil Sands

by

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Abstract

Sustainable pavement infrastructure is an essential component for the economic development for any country. The quality of materials of pavement layers impacts the long-term performance of pavement structures. Low quality granular materials can result in pavement deformation and reduced pavement life. But due to the unavailability or high cost, it is not always possible to provide good quality granular materials in the flexible pavements. In this context, stabilization have become popular in recent times to enhance the unbound layer properties. However, the commonly used stabilizing agents have some economic constraints and environmental concerns. Hence, researchers are continually seeking alternative and creative materials.

Tailing solvent recovery unit (TSRU) tailings obtained from oil sands bitumen are waste materials with no significant use in current practice. On the other hand, bitumen froth is another by-product produced in oil sand industries. The TSRU tailings stream mainly contains water, asphaltenes, fines, solids, bitumen and residual solvent. Typically, the recovered bitumen froth contains water and solids in it. In terms of economic prospect and composition, TSRU tailings and bitumen froth can be good stabilizing agents. There has been no publicly available study so far about the application of TSRU tailings and bitumen froth in pavement construction. Hence, this study aims to bridge this research gap.

The objective of this research is to investigate the application of TSRU tailings and bitumen froth in pavement construction, and reduce the thickness of granular layer of pavement by improving the layer property with these modifiers. At the initial stage of it, the characteristics of TSRU tailings, bitumen froth and granular materials were evaluated using a number of laboratory testing methods. For mixture preparation, different bitumen froth contents were added to the mixture, and different properties of the mixes were determined. To investigate the tensile strength and moisture sensitivity properties of the modified mixtures, indirect tensile strength (ITS) test was performed on modified mixtures. To estimate the change in thickness before and after TSRU modification, California bearing ratio (CBR) and Marshall stability tests were performed, and the layer thickness change was estimated according to AASHTO 1993. To improve the moisture sensitivity property of the modified samples, cement was added as an additive, then ITS test was conducted, and tensile strength ratios (TSR) were calculated for the cement-treated samples. Additionally, in order to understand the cracking resistance of cement-modified mixtures, cracking tolerance (CT) of the samples was determined using the indirect tensile asphalt cracking test (IDEAL-CT).

Results from the study indicated that TSRU tailings and bitumen froth modification could improve the tensile strength of mixtures, and reduce the thickness of the granular layers significantly. On the other hand, the moisture sensitivity of the TSRU-modified sample was found high. Cement treatment can improve the moisture resistance of the TSRU-modified samples but at the same time, it increases the cracking potential of the modified mixes.

Preface

This research was carried out at the University of Alberta's Asphalt and Binder Engineering Laboratory, under the supervision of Dr. Leila Hashemian. Dr. Leila Hashemian was the supervisor of the study and was involved in idea development during the study. I was in charge of all major aspects of the study including data collection, lab experiments, data analysis, and manuscript preparation. Muhammad Misbah Uddin, Farshad Kamran, Mohamed Saleh, and Liniker Lettiere Ferreira Monteiro assisted me in some testing. Dr. Leila Hashemian and Dr. Taher Baghaee Moghaddam assisted with manuscript revisions.

Chapters 1 and 2 of the thesis consist of the introduction and literature review which are written based on previous academic findings of relevant research works. Chapter 3 explains the materials used in the research and the properties of them. A version of chapter 4 has been submitted for publication as S. R. Sheonty, M. Saleh, F. Kamran, T. B. Moghaddam, L. Hashemian, "Performance Evaluation of Stabilized Base Courses Comprised of TSRU Materials and Bitumen Froth Derived from Alberta Oil Sands." in the Canadian Journal of Civil Engineering (CJCE). A version of chapter 5 has been submitted for publication as S. R. Sheonty, T. B. Moghaddam, L. Hashemian, "Investigation of Moisture Sensitivity of Granular Base Course Materials Comprised of Tailing Solvent Recovery Unit and Bitumen Froth Derived from Alberta Oil Sands." in the International Journal of Pavement Research and Technology (IJPRT). Chapter 6 gives the summary and conclusions of this study.

Acknowledgements

At first, I would like to express my sincere gratitude and thanks to almighty Allah. This thesis is dedicated to my loving parents for their unwavering support, encouragement, and sacrifices.

I would like to thank my supervisor, Dr. Leila Hashemian, without whom I would not have been able to complete this research. She helped during the whole research process from idea generation to articulating the research methodology. Her proper guidance, friendly attitude and constant motivation has made my journey a memorable one. I was highly inspired by her leadership quality and success in STEM field and she was an epitome of women empowerment for me. She has been the best supervisor I could ever ask for.

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Chapter 1 Introduction

1.1 Background

Comfortable and safe transportation of people and goods is one of the most important prerequisites for economic growth. With the growing civilization, the traffic demand is increasing day by day around the world. There is no alternative of having resilient and sustainable pavement structures to fulfil the increasing traffic demand. Hence, civilization is highly dependent on the pavement industry for commercial and personal purposes. Pavements are generally designed to sustain traffic loads during their intended service life period. A pavement structure consists of different layers top of one another over the ground. The resistance of such pavements to climatic conditions and transmit traffic loads depends on their layer thickness or quality of materials. There are two main categories of pavements such as: rigid (concrete) and flexible (asphaltic) pavement [1]. The most widely used pavement is flexible pavement due to its comparatively good resistance to temperature variations, good performance during its service life, high driving comfort, safety, low initial construction cost and easy maintenance [2]. Canada is one of the countries having an extensive network of paved roads. It was estimated that flexible pavements made of asphaltic materials represent 90% of the total paved road length in Canada. Flexible pavement consists of different layers that helps to transmit load. The main layers of flexible pavement are subgrade, subbase course layers, base course, and surface course layers. These layers are normally built on a suitable layer called subgrade, which finally receives and transmits traffic loadings. Amongst these layers, the base layer is an important part of the pavement structure, which plays an important role in distributing the load to subgrade. Flexible pavements normally consist of an asphalt concrete layer placed over a base and/or a subbase layer which are supported by a compacted soil called subgrade

[3]. Generally, base course is constructed with a dense graded aggregate structure, this gradation used can be made of crushed stone, crushed slag, or other untreated or stabilized materials [4].

A pavement base layer should have adequate resistance to deformations and fatigue cracking due to repeated application of loads and thermal cracking due to pavement exposure to extreme variations of temperature. Furthermore, the base course layer under traffic loading most possesses adequate resistance against distortion effects and should be able to prevent moisture induced damage [5]. Seasonal changes in temperature or excessive traffic loading cause distresses on flexible pavements. The prominent distresses for flexible pavements are permanent deformation, rutting or cracking [6]. Cracked pavements are sensitive to continuous freeze and thaw cycles due to water ingression [7]. Hence, the distresses can cause a significant reduction in the service life of the asphalt pavement. Regular maintenance and prevention planning can play an important role in extending the service life of pavements, especially in cold regions such as Canada. The governments and agencies spend huge amounts of money on pavement maintenance, reconstruction, or rehabilitation on it. The traffic load is also increasing day by day as a result of growing economy of the world, meanwhile, extreme climatic conditions has increased due to climate change. Both the factors can increase the rate of pavements deterioration around the world. Therefore, it has become necessary to improve pavement layers to prevent premature distresses in asphaltic pavements. A typical solution of mitigating pavement failures is increasing the thickness of base course layer. But it is not economically viable as it increases initial construction cost. Usually, the quality of granular material has a direct relationship with the load bearing capacity of the pavement layers. But due to the unavailability or high cost, it is not always possible to provide good quality granular materials in the flexible pavements. In this context, stabilization of base course by adding different stabilizing agents have become a good alternative in recent times to enhance the unbound layer properties.

Stabilization techniques can improve the performance of the pavement base course layer. The two main techniques of base course stabilization are mechanical stabilization and chemical stabilization. Mechanical stabilization is a method that improves soil properties by grading the soil with compression and densification method by using mechanical energy like rollers, rammers, and vibration techniques [8]. Meanwhile, chemical stabilization modifies soil properties by mixing or injecting chemically active compounds such as Portland cement, lime, fly ash, or viscoelastic materials [9,10]. Common materials used for chemical stabilization are asphalt emulsion and active fillers such as Portland cement. Recent studies have shown that stabilization of base materials increase the tensile strength, stability, bearing capacity, and mechanical properties of the layer [9,10]. According to Wirtgen Cold Recycling Manual, different forms of asphalts such as cutback, foamed, and asphalt emulsion, and cementitious materials including hydrated lime, cement, fly ash can be used effectively for stabilization depending on the characteristics of the soils to be treated [7]. Previous studies suggest cementitious materials improve the performance properties of stabilized asphalt emulsion mixtures and increase the curing rate of asphalt emulsion stabilized mixtures [11,12]. However, the using cement in stabilized mixtures has several drawbacks such as high cost and the risk for thermal cracking. Apart from that, a large quantity of energy is needed during cement production which produces significant CO_2 [13]. On the other hand, as per the study of Fang et al., asphalt emulsion has comparably low energy consumption and pollution [14]. But the asphalt emulsion needs a long curing time to achieve binding properties [11] which results in lower mechanical strength at the early stages of its life [15].

In recent times, the high thickness of some roads such as mine road is a matter of concern. In order to reduce the thickness and improve the quality of materials of the layers, new stabilizing agents are needed to overcome the drawbacks of commonly used stabilizing agents. Hence, continual research has been conducted to find alternative and creative materials that can overcome the economic limits and environmental concerns. In this context, tailings solvent recovery unit (TSRU) tailings and bitumen froth (BF) can be interesting and cost-effective materials to be used in the pavement industry to stabilize base course. They are by-product materials produced during bitumen production from oil sands. The TSRU tailings stream mainly contains water, asphaltenes, fines, solids, bitumen and residual solvent (about 5 to 10%) [16]. As per previous studies regarding asphaltenes [17,18], it is expected that addition of TSRU materials can potentially improve the performance properties of stabilized asphalt mixtures due to the presence of asphaltenes and organic matter in it. Typically, the recovered BF consists of 60 wt% bitumen, 30 wt% water, and 10 wt% mineral solids [19, 20, 21] where the treated BF can still contain 2 wt% to 5 wt% water and 0.5 wt% to 1 wt% solids on average [22,23]. There have been no specific studies publicly available till now on the application of TSRU materials and bitumen froth as stabilizing agents in the pavement industry. In this scenario, this study focuses to fulfil this research gap and try to investigate the application of TSRU tailings and bitumen froth in pavement construction.

1.2 Objectives

Currently, there is no application of TSRU tailings and bitumen froth in any industry. On the contrary, there is a demand of finding innovative and cost-effective stabilizing agents to improve granular material properties of pavement layers. In this context, the study focuses to investigate the application of TSRU tailings or bitumen froth in pavement engineering. The specific objectives of the study are as follows:

- To improve the granular material properties and reduce the thickness of granular layers with no additional costs by using a waste material, i.e., TSRU tailings along with bitumen froth, as stabilizing agents.
- To investigate the impact of TSRU modification to improve the tensile strength performance of the modified mixtures using indirect tensile strength (ITS) test.
- To determine the change in thickness of granular layer after TSRU modification by conducting California bearing ratio (CBR) and Marshall stability tests.
- To improve the moisture resistance property of TSRU modified mixtures by using Portland cement as an additive and compare moisture sensitivity of modified samples before and after cement addition using tensile strength ratio (TSR).
- To investigate and compare the cracking resistance of cement-treated mixtures with untreated mixture by the indirect tensile asphalt cracking test (IDEAL-CT) analysis.

1.3 Methodology

At the initial stage of this research, comprehensive laboratory tests were conducted to evaluate the physical properties of the TSRU tailings, bitumen froth and granular material as the properties of these materials used in this study was unknown. After evaluating the materials properties, TSRU tailings and bitumen froth were used to prepare mixture at optimum moisture content (OMC) of granular materials. The results obtained from different performance tests were used for assess the suitability of TSRU modification in pavement engineering. For investigating the tensile strength property, mixtures were prepared with both aggregate samples, bitumen froth and TSRU tailings at different bitumen forth content and ITS tests were conducted on them. From the ITS test, the optimum bitumen froth content was found. The impact of TSRU tailings in improving the tensile strength property was analyzed comparing the ITS test results to unmodified samples. The change in thickness of granular layer after TSRU modification was calculated from the Marshall stability and CBR test results. In order to improve the moisture resistance, Portland cement was added as an additive and cement-treated samples were prepared in different cement content. The impact of cement in improving the moisture resistance and tensile strength was analyzed. Moreover, the cracking resistance of the cement-treated samples was analyzed with IDEAL-CT analysis. The methodology of the research is shown in Figure 1-1.

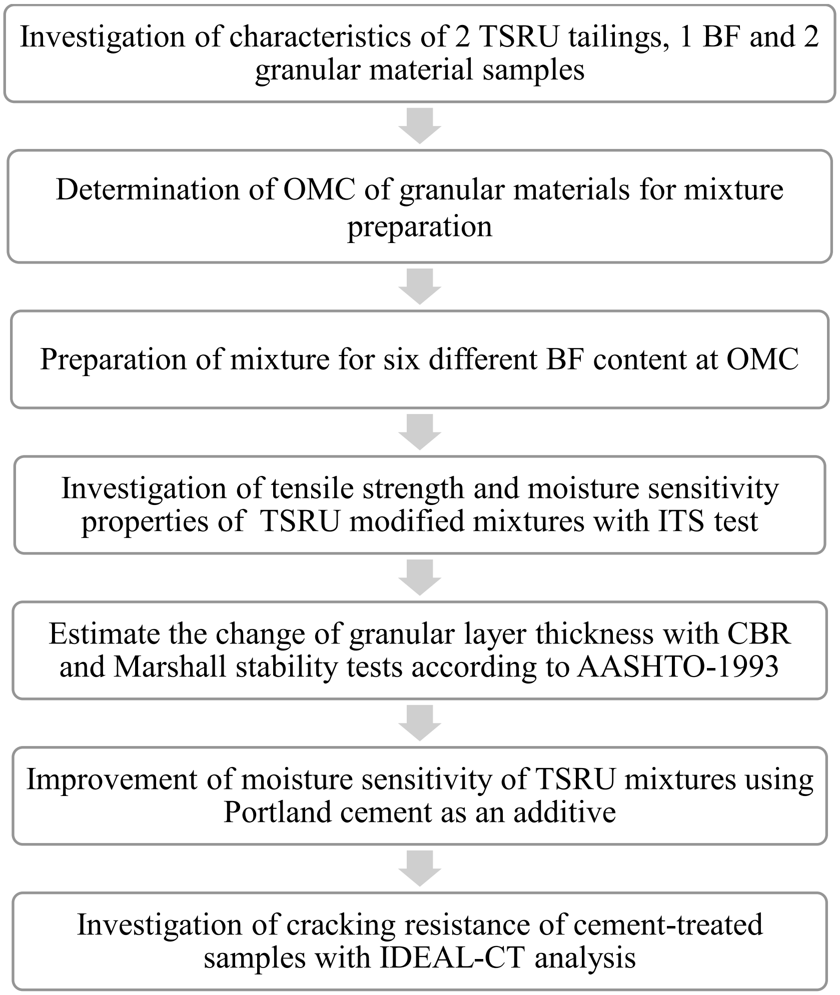

Figure 1-1 Schematic diagram of methodology of the research

1.4 Thesis Outline

This thesis is organized in five chapters and presented as follows:

Chapter 1 – Introduction: In this chapter, the background of the research work along with the objectives, methodology and thesis structure are presented

Chapter 2 – Literature Review: In this chapter a detailed review on the asphalt stabilized mixtures and description about TSRU tailings, bitumen froth is presented. Different mixture procedures, production and treatment of TSRU materials and bitumen froth and previous studies are discussed in this section.

Chapter 3 – Materials: In this chapter a detailed description on the properties of the materials used in the study is presented. The properties of TSRU tailings, granular materials and bitumen froth are discussed in this section.

Chapter 4 – Performance Evaluation of Stabilized Base Courses Comprised of TSRU Materials and Bitumen Froth Derived from Alberta Oil Sands: This chapter evaluates the effects of TSRU modification on asphalt stabilized mixes by conducting ITS test. Additionally, the change in granular layer thickness is discussed before and after TSRU modification by conducting Marshall stability and CBR tests.

Chapter 5 – Investigation of Moisture Sensitivity of Granular Base Course Materials Comprised of Tailing Solvent Recovery Unit and Bitumen Froth Derived from Alberta Oil Sands: This chapter investigates the moisture sensitivity of TSRU modified samples and tried to improve this property by using cement as an additive. Moreover, a comparison is shown between cement-treated and untreated samples to understand the impact of cement treatment to improve moisture resistance of TSRU modified samples. Additionally, the cracking resistance of cement-treated samples is also discussed on this chapter.

Chapter 6 – Summary and Conclusions: In this chapter, the performance of TSRU modified mixes are summarized and explained based on laboratory tests and observations. In addition, this chapter summarizes the idea, objectives, and scopes of the thesis.

Chapter 2 Literature review

2.1 Asphalt Mixtures

The major function of the base course layer is to provide the load-supporting capacity through load distribution to the other pavement layers. Hence, this layer needs to have adequate resistance against permanent deformation, fatigue cracking caused by repeated loading, and thermal cracking when being exposed to low temperatures or intense temperature fluctuations. For this reason, the base course has a dense graded aggregate structure, which can be composed of crushed stone, crushed slag, or other untreated or stabilized materials [7]. Different asphalt mixtures including hot mix asphalt (HMA), cold mix asphalt (CMA) and warm mix asphalt (WMA) can be used for pavement construction especially in base layers. Usually, HMA consists of aggregate and a viscous binding agent, and is produced at a temperature range of about 300 to 350°F [24]. There are three subcategories of HMA: dense mixes, stone matrix asphalt, and open grade mixes. Dense-graded mixes are often used for high traffic roadways due to its durability, while, stone matrix asphalt is produced to improve tire grip and prevent rutting on roads. This mix contains more asphalt cement, while also adding binders and fibers to the mix. On the other hand, WMA is produced at a temperature range of 200-250°F [24]. Some typical WMA technologies include foaming effect, organic additive, and chemical package. Addition of water helps to lower the temperature and expand the volume of the asphalt binder, finally results in creation of foam. The foaming effect increase workability of the mixture. The organic additive can be used to avoid permanent deformation because of its ability of reducing the asphalt viscosity and improving the flow. The chemical package can be regarded as a compaction aid that can improve the asphalt's workability, emulsification, and adhesion. Lastly, cold mix is the type of mixture that doesn't need heat during the mixing process. These types of are composed of asphalt emulsion and unheated aggregates which are mixed and compacted at ambient temperature, that no heating is required for its preparation [25,26]. Usually, it is used to repair cracks over an inch wide and potholes that pop up during the winter months. It cannot be considered as a substitute for a formal repair with hot mix or warm mix asphalt during the warmer months.

Comparing the advantages and disadvantages of the mixtures of HMA and WMA, it can be stated that HMA is reliable due to its durability, strength, and low cost. Moreover, its installation process is easy and simple that takes only a short period of time until it can be used. It also generates less traffic noise comparing to the pavement made by other mixtures and is rough enough to prevent skidding in wet conditions. HMA is recyclable which limits natural resources used in engineering projects, promoting sustainability in the field. Though the quality of the mix is good but it is more expensive than other types of asphalt mixtures, hence, it is recommended to use on large asphalt projects [24]. Meanwhile, WMA offers a suitable balance between HMA and CMA. The goal of using WMA is to produce quality dense asphalt mixtures but at lower temperatures which will cost-effective without compromising the quality. The purpose of producing asphalt at lower temperatures is to reduce emissions and boost energy savings and it can save up to 30% more energy compared to HMA. The emission reduction makes it environmental-friendly and less temperature makes it worker friendly. It also can provide better compaction of pavements, a longer fatigue life, and the possibility to use more recycled material. There are also some construction benefits associated with WMA which includes a longer paving season in colder regions and the potential for longer hauling distances [24]. Disadvantages associated with WMA are still under research today. The long-term performance of WMA is uncertain, and the possibility of moisture sensitivity is greater than that of HMA [24]. It can potentially result in more frequent maintenance and rehabilitation of pavements.

In this context, CMA offers some extra advantages compared to HMA and WMA. CMA does not require heating like conventional HMA or WMA. CMA has the advantage that, unlike hot mix asphalt, these types of mixes can be produced at both work site or in the plan. It helps to reduce the cost of hauling as well as a significant reduction in energy consumption as well. Additionally, a large proportion of recycled asphalt pavement (RAP) can be used in the cold mix, these actions significantly reduce the cost of material for pavement construction. Hence, this type of mix is very cost-effective comparing to the other mixes [25]. The general application of cold mix asphalt mixes is mostly in a base course under conditions of low or medium traffic specially in cold climatic regions. Even though some benefits were recorded in the application of cold mixes, some challenges or drawbacks were also found to be associated with cold mixes, prominent among these drawbacks are the high porosity of the compacted mixture, longer curing time before achieving maximum strength, or weak early life strength [26, 27,28]. In this study, CMA technique has been chosen because of the advantages and cost-effectiveness of this technique.

2.2 Stabilization Techniques

In flexible pavements, repeated loading on unbounded granular base courses results in densification which subsequently leads to deformations over a period of time [29]. Thus, it is necessary to construct a granular base course with high-quality material to increase the resistance of the layer under loads conditions during service life. But due to the high cost and unavailability of these materials, it is not always possible. In this scenario, a suitable alternative is stabilization of base course using different stabilizing agents to increase the strength of the layer. The main purpose of the stabilization technique is to increase the stability, strength, bearing capacity as well as other performance properties of a base course layer. Numerous performance properties including shear strength, stiffness, durability, and moisture resistance can be improved through base course material stabilization techniques [30].

Two techniques of base course stabilization are mainly used in the industry. They are mechanical stabilization and chemical stabilization. Mechanical stabilization improves soil properties by grading the soil with compression and densification method by using mechanical energy like rollers, rammers, and vibration techniques [8]. On the other hand, in chemical stabilization, soil properties are modified by mixing or injecting chemically active compounds such as Portland cement, lime, fly ash, or viscoelastic materials [9,10]. The chemical components those are used in this stabilizing process are known as stabilizing agents. A wide range of stabilizing agents are used in this world such as wetting agents (i.e., surfactants for example sulphonated oils), hygroscopic salts (for example calcium chloride), natural and synthetic polymer, modified waxes, petroleum resins, bituminous materials (for example asphalt emulsions), cementitious materials (for example fly ash, Portland cement, lime) [7]. The aim of the stabilizing agents is to bind the individual aggregate particles together to increase strength, stiffness and durability. Proper type of stabilizing agents and its content is important for getting desired result after stabilization. Different soil material requires different stabilizing agent or additive depending various soil properties for achieving the desired properties of the pavement layers. For example, a lime material is usually added between 1% to 4% of the total mix, Portland cement can be added within a range of 1% to 3% of the total mix, and fly ash material should be added between 6% to 20% of coarse aggregates weight for the optimum result [9].

Among the stabilizing agents, bituminous and cementitious materials are the most commonly used stabilizing agents in the world [7] and there have been many studies which indicates cementitious materials can improve different performance properties of stabilized asphalt emulsion mixtures

[11,12]. However, the use of Portland cement in stabilized mixtures has several drawbacks such as high cost and increased risk of thermal cracking. In addition, it is not environment-friendly because a significant volume of CO_2 is released into the atmosphere during cement production [13]. On the other hand, asphalt emulsion has comparably low energy consumption and pollution [14]. But the asphalt emulsion needs a long curing time to achieve binding properties [11] which results in lower mechanical strength at the early stages of its life [15]. Given these economic constraints and environmental concerns, researchers are continually seeking alternative and creative materials. In this context, investigation of bitumen waste materials (i.e., TSRU tailings) and bitumen froth as stabilizing agents can be an interesting research topic in the field of pavement engineering. Hence, TSRU tailings and bitumen froth have been used in this study to stabilize the base course.

2.3 TSRU Tailings

During paraffin-based froth treatment process, a tailings stream is generated that consists of fine solids, water, asphaltenes and trace amounts of bitumen and solvent. In the froth treatment plant, paraffinic solvent is added to froth with an objective of separating bitumen from water and solids. The water and solid tailings from the froth treatment plant are sent to the tailings solvent recovery unit to recover the paraffinic solvent. Tailings solvent recovery units are used to strip-off and recycle any residual solvent, prior to disposing the tailings in a storage pond. Once the tailings are processed by the solvent recovery unit, they are known as TSRU tailings. The tailings stream mainly contains water, asphaltenes, fines solids, bitumen and residual solvent. Solvent content in the tailings can vary between 5 and 10%, although the exact composition of the tailings stream varies according to operating conditions [16].

2.3.1 Production of TSRU Tailings

TSRU tailings are generated during the bitumen production process as a by-product. But the tailings are processed by solvent recovery process. It is a mechanical process where modified steam-stripping flash columns are used to execute the process. Using cooling water solvent is vaporized, condensed inside these columns and recycled back to the front end of the paraffin-based froth treatment process. Due to the presence of asphaltenes, there are a few variations on the TSRU flash-column design used in the oil sand industries. There are 2 types of columns currently in service: a flash column with agitator and little internals, and a flash column with internal shed decks and no moving parts [31].

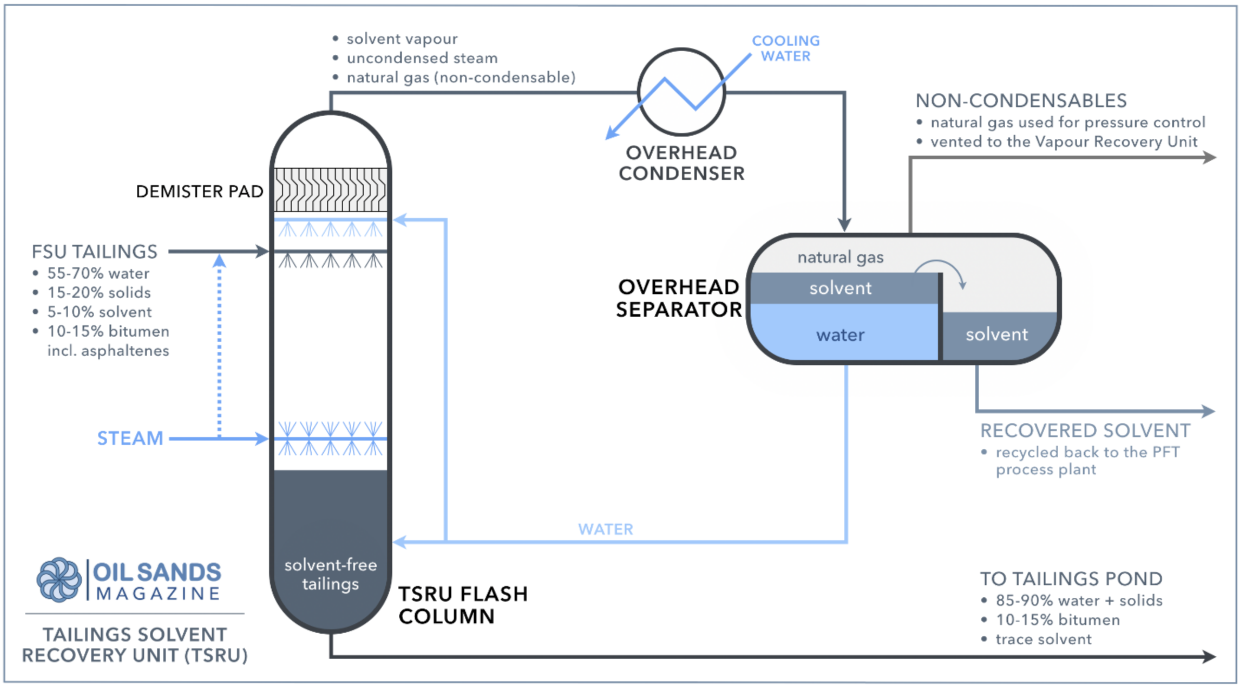

Figure 2-1 Schematic diagram of TSRU production process [31]

TSRU column is almost identical to the naphtha recovery unit (NRU) flash-column used in naphthenic froth treatment (NFT) facilities. Feed from the second stage froth settling unit (FSU) underflow is diluted with hot water and pumped to the top of the column, where the slurry is distributed over a series of shed decks. Steam is introduced into the bottom of the vessel, just above the liquid level. As steam rises up the column and contacts the tailings counter-currently, the solvent is stripped out of the tailings. The shed-decks help spread out the tailings and improves contact between the tailings and the steam. Demister pads at the top of the column capture any fines that might get carried up into the overhead system. On the other hand, the TSRU design used by Shell has no internal shed-decks but instead relies on agitation to improve steam recovery. Steam is injected directly into the tailings prior to being fed into the vessel. This induces a pressure drop and atomizes the tailings as it enters the column. The liquid pool is simultaneously agitated using an impeller, adding more shear to the system which improves solvent recovery. The agitator stirs up the asphaltenes, breaking up agglomerations and reducing foaming. If foam is detected in the TSRU column, that foam can be redirected to a foam-breaking vessel, which operates at a lower pressure than the columns. The foam is further agitated and sprayed with hot water and defoamer chemicals. The de-foamed liquid is then pumped back to the main TSRU flash columns [31].

To improve solvent recovery, TSRU columns are typically arranged in pairs, normally operated in series. However, high-shear environments have been found to greatly improve the liberation of solvent from the asphaltene agglomerates. TSRU columns are normally equipped with bottoms recycle pumps that can recirculate the underflow back into the vessel which improves solvent recovery, reduce foaming, prevent the formation of asphaltene mats and prevent plugging of the column during upset conditions [31]. Asphaltenes contain natural surfactants which can cause severe foaming within the TSRU vessels. Foaming can cause fine solids to be carried-over into the overhead system and can also cavitate the underflow pump. Foaming within the TSRU column can be mitigated by adding hot water steam or applying shear to the slurry through mixing or pumping and adding chemical defoamers and asphaltene dispersants. Hot dilution water is commonly added to TSRU vessels in order to reduce foaming, however this has been met with very limited success. The problem associated with it is the large volumes of hot water which is required to suppress the foam. Defoamers and other chemicals are usually added to the TSRU circuit to more effectively mitigate foaming [31].

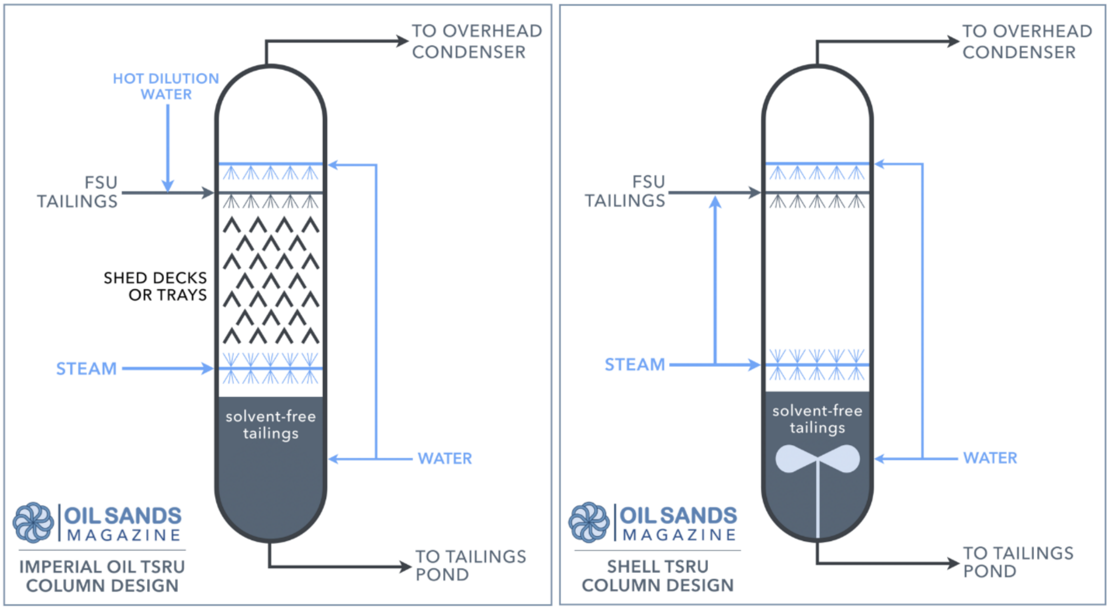

Figure 2-2 Types of columns used in TSRU production process [31]

The operation and control process of TSRU tailings production is important. The liquid level in the TSRU flash column is usually measured by differential pressure and controlled by the underflow tailings pumps where the level in the column is usually maintained below the steam injection header. Steam injection is added at a fixed flowrate or as a ratio of the feed stream. Nuclear density meters or densitometers can be installed at various intervals to detect foaming in the column and defoamers and other chemicals are added on an as-needed basis. The water interface level in the overhead separator is typically measured using differential pressure and the interface is controlled by adjusting the outflow of the water, using a control valve or pump. Water is commonly recycled back into the TSRU column. The solvent liquid level in the overhead separator is also normally measured using differential pressure and is controlled using a control valve or pump, which directs the solvent back to the front end of the process. Both the TSRU flash column and overhead separator operate at the same pressure since they are physically connected. Pressure of the system is controlled by a pressure control valve which relieves non-condensable to the vapor recovery unit or by adding natural gas or nitrogen to the separator. Some facilities operate the system under vacuum instead of near-atmospheric pressure where a vacuum pump is required to maintain the negative pressure [31,32].

2.3.2 Treatment of TSRU Tailings

TSRU can contain residual paraffinic solvent, other hydrocarbons, and sulphides. Hence, because of potential environmental risks associated with TSRU tailings, the treatment of TSRU tailings is needed before disposal of it in the disposal ponds. During treatment process, the solvent must be recovered before the tailings can be stored in the tailings pond. TSRUs used in the oil sands are modified versions of traditional flash columns, using temperature and pressure to recover the solvent. The solvent-free tailings can then be stored in the tailings pond for future reclamation.

2.3.3 TSRU Tailings as Stabilizing Agent or Modifier

TSRU tailings stream contains organic matter including asphaltenes [16]. Asphaltenes can be obtained from different sources including oil sands, crude oil, asphaltite, tar sand, and bituminous coal. Previous literatures indicate that the application of asphaltenes can improve the rheological properties of asphalt mixture. The study by Kamran et al. indicates that the high-temperature properties of modified mixtures improved significantly in comparison with the unmodified ones after using asphaltenes in the asphalt emulsion stabilized mixtures [17]. Basavarajappa et al. concluded that the addition of asphaltenes to asphalt emulsion increased the shear modulus of the asphalt emulsion base binder and as a result improved the modified mixture stiffness and rutting resistance [18]. The results previous studies indicated that the addition of TSRU tailings to asphalt mixtures will potentially improve the properties of stabilized asphalt mixtures due to the presence of asphaltenes in TSRU tailings.

However, TSRU tailings obtained from oil sands are considered as a waste, this material has no significant applications in the industry. On a contrary, TSRU tailings are produced at a higher rate in the oil sand industries. Hence, disposal and management of TSRU tailings is a big concern for the oil sand industry. TSRU tailings are typically disposed in large tailings ponds after treatment which involves high cost, time and energy. Thus, using TSRU tailings in pavement construction will also be a very cost-effective solution as it will minimize the cost of tailings pond management as well. From both economic perspective and the composition of TSRU tailings indicate its potential as a pavement construction material. This study regarding application of TSRU tailings in road industry is important because apart from enhancing the properties and cost performance of the stabilized mixture, it can identify a good use of waste material (TSRU tailings) and also minimize the disposal and management hassles.

2.4 Bitumen Froth

Bitumen froth is a by-product which is generated from bitumen recovery process from oil sands. It contains bitumen, water and some amounts of solids. Due to the composition and presence of organic matter in it, it is expected to be a good substitute of asphalt emulsion for mixture preparation.

2.4.1 Bitumen Froth Composition

Usually, BF has of bitumen, water and some solids. The recovered BF consists of 60 wt% bitumen, 30 wt% water, and 10 wt% mineral solids [19]. A froth treatment is necessary to separate the water droplets and mineral solids from the organic bitumen product using hydrocarbon solvents, although the micrometer-sized mineral particles (mainly clays) and water-in-oil emulsion droplets present in BF are difficult to remove [20]. Naphtha is added as a diluent to the bitumen froth in order to decrease its viscosity and to liberate the hydrocarbon components from the inorganic contaminants (mineral solids and water). The fine mineral particles are the main detriments in stabilizing the water-in-oil emulsions, where emulsified water droplets are easy to destabilize and remove in the absence of fine mineral particles [19,20]. After treatment, the water and solid content decreases, the treated BF can still contain 2 wt% to 5 wt% water and 0.5 wt% to 1 wt% solids on average [22,23].

2.4.2 Bitumen Froth Extraction

The oil sands ores are mined using open pit surface mining technology, crushed to break the lumps, and mixed with water (50−80 °C). The formed slurry is transported using hydrotransport pipelines to primary separation vessels where bitumen is recovered by flotation as a bitumen froth. The transporting pipelines also serve the purpose of conditioning the oil sands so that they are ready for separation in the PSV. [20, 21]. An alternative bitumen extraction process, mainly suitable for deep-buried oil sands deposits, is the in-situ technology, such as the steam-assisted gravity-drainage (SAGD) technique. In the SAGD technique, steam is injected into upper horizontal wells to heat the oil sands, and the mobilized bitumen drains into the lower horizontal wells where it is produced. The fluid recovered from SAGD is also a mixture of bitumen, water and mineral solids, called bitumen emulsion [21, 22].

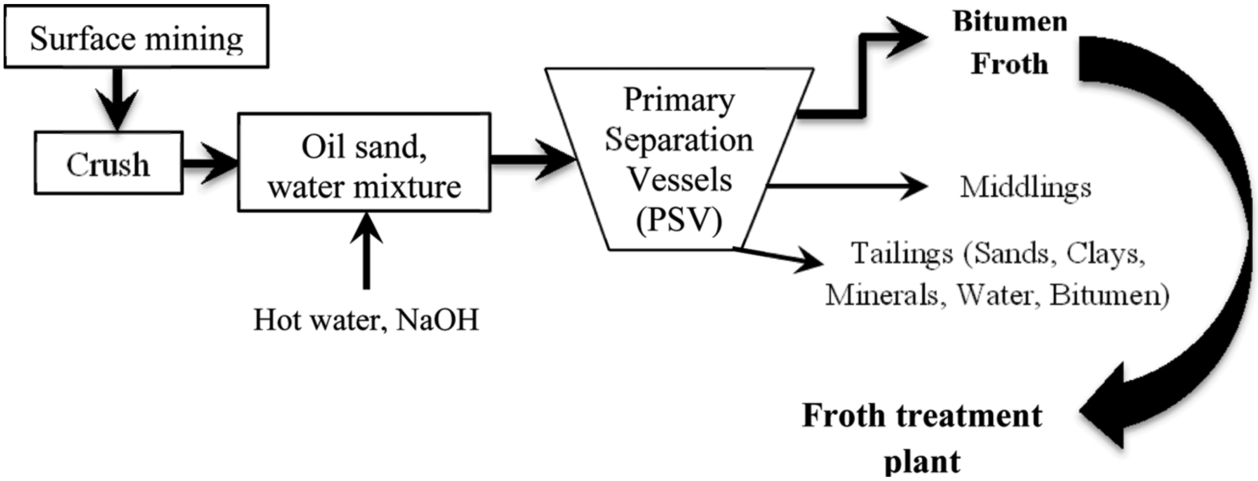

Figure 2-3 Schematic diagram of extraction of bitumen froth [22].

2.4.3 Bitumen Froth Treatment

Bitumen froth treatment is an important step in the oil sands bitumen recovery operations. The objective of this step is to separate mineral solids and water from the bitumen froth. The bitumen froth is diluted with naphthenic or paraffinic solvents to lower its viscosity. Mainly it facilitates the separation of bitumen froth treatment is the removal of inorganic (mineral particles and water droplets) from a bitumen organic solvent solution. The water-in-oil emulsions are formed by water entrained into the bitumen froth during the water-based extraction process and stabilized by natural surfactants in bitumen (especially asphaltene) and fine mineral particles. In fact, the fine mineral particles are the main detriments in stabilizing the water-in-oil emulsions, for the emulsified water droplets were found to be easy to destabilize and remove in the absence of fine mineral particles. Effective removal of the fine mineral particles and water droplets requires that the fine mineral particles to form larger aggregates. Moreover, the water-in-oil emulsions should be destabilized.

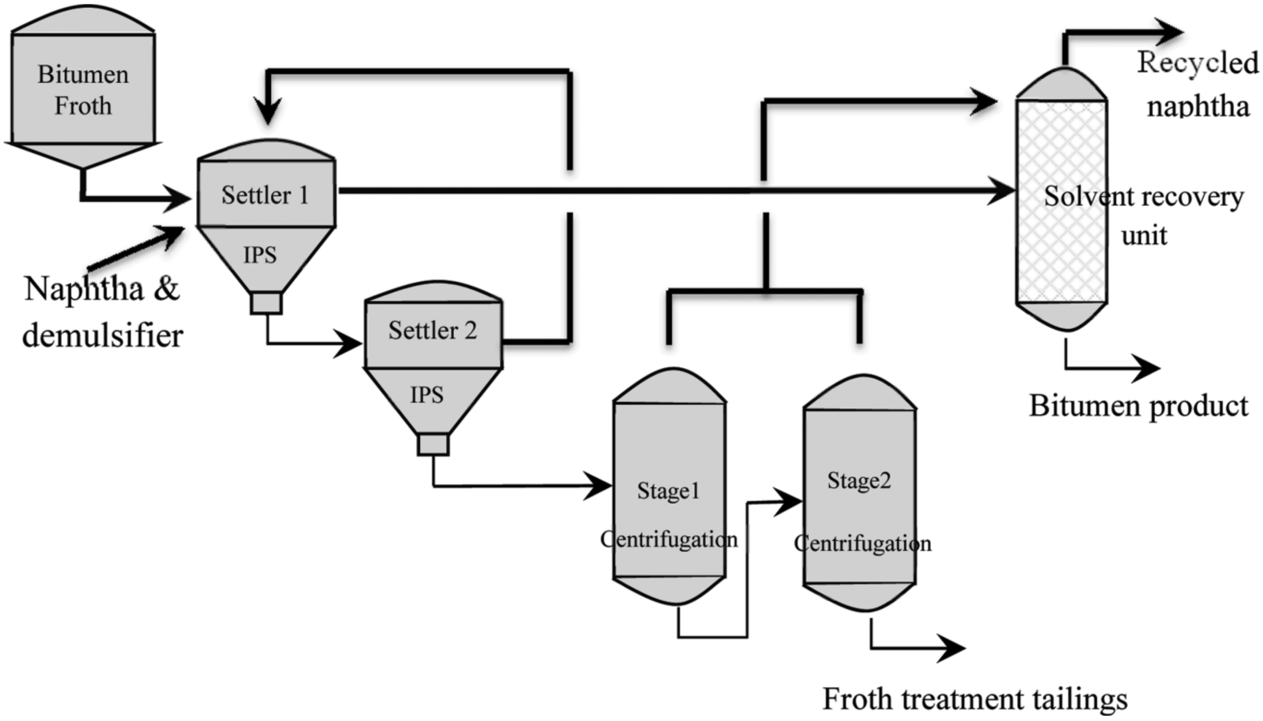

Figure 2-4 Schematic diagram of treatment process of bitumen froth [22].

Multistage centrifugation (with centrifugal forces up to 2500 G) and the addition of demulsifies are applied for the removal of solids and water in-oil emulsions from the diluent-bitumen solutions. The treated bitumen froth can still contain on average 2−5 wt % water and 0.5−1 wt % solids [22].

2.4.4 Bitumen Froth as a Binder

Previous literatures proved that the addition of asphaltenes can improve the rheological properties of asphalt mixture. Basavarajappa et al. [18] proved that the addition of asphaltenes to asphalt emulsion can increase the shear modulus of the asphalt emulsion base binder and it can improve the stiffness and rutting resistance of the modified mixtures. The high-temperature properties of modified mixtures can be improved significantly after adding asphaltenes in the asphalt emulsion stabilized mixtures [17]. Bitumen froth contains organic matter including asphaltenes [22]. Hence, due to the presence of asphaltenes, the addition of TSRU tailings might improve the properties of stabilized asphalt mixtures.

2.5 Portland Cement as an Additive

Portland cement can be used as an additive to the cold recycling asphalt mixtures. Previous studies indicated that it helps to improve various properties including stiffness, rutting depth, permanent deformation and moisture resistance [33, 34, 35]. A study by Xu et al. [33] investigated the impact of 0.5%, 1.5%, and 2.5% cement contents on moisture resistance, rutting and high- and low-temperature properties. Various performance tests were conducted such as ITS in both dry and wet condition to determine the moisture resistance ability, wheel tracking test to assess rut depth and dynamic stability for high-temperature stability, and bending beam test to evaluate low-temperature performance. ITS test results showed that after moisture subjection, cement plays a positive role in resisting moisture damage. From the wheel tracking test, 1.5% was the optimum cement content as it provided the maximum value for dynamic stability and lowest rutting depth. The study further elaborates that specimen with 2.5% cement leads to poor workability due to excessive stiffness for compacting. Bending strength at failure and bending strain at failure are evaluated based on elementary beam theory from the three-point bending beam test. The results highlighted that bending strength at failure increased, while the bending strain at failure decreased with the increase in the amount of cement. The study suggested that failure in cold recycled asphalt mixtures is due to excessive strain and high content of cement.

A study by Niazi and Jalili [34] investigated the effect of active fillers such as Portland cement, hydrated lime slurry (HLS), and hydrated lime (HL) and compared the results among the active fillers. Upon performing ITS dry and wet tests, it was found that samples without the active fillers were sensitive to moisture damage and the tensile strength ratio (TSR) of the sample with the

Portland HLS provided the best result followed by Portland cement and HL. The permanent deformation of the cold recycling asphalt mixtures was also improved with the introduction of active fillers as found from the dynamic creep test. It was discovered that the Portland cement, HLS, and HL resulted in a reduction of rutting depth by 40%, 30%, and 26% when compared to the sample without the additives. Furthermore, the wheel tracking test also emphasized a similar trend where Portland cement, HLS, and HL resulted in a decrease of rutting depth by 58%, 50%, and 38% when compared to the sample without any fillers. Based on the experiment, it is evident that either Portland cement or HLS could be highly effective for application in the cold recycling asphalt emulsion mixtures. Although both of these active fillers are beneficial, due to the difficulties in producing hydrated lime slurry the study reports that the use of Portland cement is recommended. Furthermore, the study shows that a cement content between 1% to 2% is the optimum cement content for significant improvement in performance properties of the mixes.

In a study by Yan et al. [35] a number of tests including Hveem cohesion test, raveling test, immersion/ freeze-thaw IDT test, wheel track rutting test, and three-point bending tests were conducted. The main focus was to understand the role of 1%, 1.5%, and 2% cement in the early-age strength and long-term performance of asphalt emulsion cold recycled mixture. Based on the results, it was found that the increase in the amount of cement results in higher cohesion force and lower raveling loss rate, which indicates that cement indeed positively contributes to the early-age strength. Furthermore, the higher content of cement also showed a trend in higher moisture susceptibility ratios for the immersion/freeze-thaw IDT test when compared to the sample without any cement. The wheel track rutting test results showed an increase in dynamic stability values with higher cement contents, suggesting that cement is advantageous for the high-temperature stability of asphalt emulsion cold recycling mixture. Lastly, the three-point bending beam test highlighted that at low temperature 1.5% cement is optimum since the failure strain percentage decreased after exceeding 1.5%. Therefore, although it is evident the addition of cement enhances the long-term performance of cold recycling asphalt emulsion mixtures when concerning about low-temperature effect, the cement content should not be greater than 1.5% according to this research.

2.6 Design of Asphalt Mixes

Asphalt mix design is divided in several steps as well as testing protocols based on the requirement for a particular application and level of traffic load intended to be used [7]. Previously, the widely used mix design methods for asphalt mixes were the Hveem design method and Marshall mix design. Hveem design method was used in California, and the Marshall mix design developed by the Illinois department of transportation. However, as there is no broadly accepted mix design currently available for asphalt warm mixes, a guideline based on empirical formulae, laboratory tests as well as previous experiences has been developed by various agencies such as Asphalt Institute [36]. Hence, in this study, prior to mix design, grain size distribution and the optimum moisture content (OMC) of the granular materials were investigated. These gradations were kept consistent for all subsequent mixtures. At first, the aggregate samples were oven-dried at 110°C for 24 hrs and crushed using a wooden hammer. Then, only the particles passing through a 20-mm sieve were selected for the preparation of the mixtures. The mixing process was initiated by adding aggregates, wet TSRU tailings in hot condition (95°C) to the blend to reach the OMC of aggregates. The total water content in the mixtures was considered the OMC of the aggregates. During the mixing process, no external water was added, rather the moisture of TSRU wet was used to reach the optimum moisture content. Thereafter, hot BF (at 95°C) was added to the mixture. The BF was heated at 95°C because of the presence of water in it. The samples were mixed until the TSRU materials and BF were uniformly distributed throughout the aggregate blend.

2.7 Performance Evaluation of Asphalt Mixtures

Using the appropriate optimum asphalt emulsion content determined, different asphalt mixes are prepared and various laboratory tests were conducted on them to evaluate their tensile strength, moisture-induced damages and cracking resistance.

This chapter is presenting the theory of the indirect tensile strength test, Marshall stability test, and IDEAL-CT analysis.

2.7.1 Indirect Tensile Strength

Indirect tensile strength test is a good indicator of assessing the tensile strength property of asphalt mixtures. The tensile properties of an asphaltic material are highly related with the cracking properties of the material. It well established that the higher the ITS value of a sample the higher the cracking resistance of the sample. The performance of the asphalt mixture to fatigue cracking is also dependent on the tensile properties. Because of repeated traffic load on the pavement layers which generates tensile stress and strains at the bottom of the pavement structure, these stresses generated leads pavements to fatigue failure during service life [37]. The stiffness of an asphaltic material determined the magnitude of the strain of a sample. Based on this the ITS test can be used as a good indicator of strength as well as adherence against fatigue failure, cracking and rutting of the asphalt mixture [37]. The effects of saturation and accelerated water conditioning of an asphaltic material can also be evaluated through indirect tensile strength test. For ITS test, samples can be prepared and conditioned in accordance with AASHTO T 283-21 [38] specification which is also known as Lottman procedure.

The indirect tensile strength test can be conducted using a universal testing machine (UTM) with Marshall samples of dimensions approximately 100mm in diameter and 60mm height. For unconditioned samples, the dry samples can be tested directly at a loading rate of 50 mm/min. During tests, the maximum load is recorded directly, and the indirect tensile strength of the sample is calculated in accordance to the equation 2-5. After unsoaked conditioning, the specimens are then conditioned by transferring in to UTM chamber at 25±0.5°C for 3 hrs. Before the soaked ITS test, the samples were conditioned in water at 25°C for 24 hrs. Finally, the samples are tested for indirect tensile strengths and the tensile strength ratios (TSR) which is a measure of resistance against moisture is calculated using equation 2-6. NCHRP Report 673 [39] suggests that the TSR of an asphaltic material should be beyond 70% in order to have adequate resistance against stripping and moisture damage.

$$S_t = (2000*P)/(\pi*t*D) \qquad [2\text{-}5]$$

Where,

S_t = indirect tensile strength (ITS), kPa

P = maximum load, N

t = average specimen thickness, mm

D = specimen diameter, mm

$$TSR = S_2/S_1 \qquad [2\text{-}6]$$

Where,

TSR = tensile strength ratio

S_1 = average tensile strength of the dry subset, kPa; and

S_2 = average tensile strength of the conditioned subset, kPa.

2.7.2 IDEAL-CT Analysis

Cracking in asphalt pavement is a major distress of asphaltic material in North America and other parts of the world. In the past, different cracking tests for asphalt mixes have been developed to ascertain the cracking damage of an asphalt material. However, the IDEAL-CT is considered as the ideal cracking test to be used for estimating the cracking resistance of asphalt material. The IDEAL-CT test is similar to the traditional indirect tensile strength test, where the test is run at 50 mm/min and at room temperature using Marshall specimens (100 mm or 4 in diameter) or Superpave (150 mm or 6 in) samples with different thicknesses (38, 50, 62, 75 mm, etc.). Samples from the laboratory are normally prepared to have air voids of 7±0.5 percent.

The cracking tolerance (CT-Index) is a parameter derived from the load-displacement curve of the samples after testing and it is used to determine sample resistance against fatigue cracking. For the cracking resistance, the higher the CT index value of the sample, the higher the fatigue resistance of the sample [40]. The IDEAL-CT is considered one of the most cost-effective and time-efficient tests for cracking due to its simplicity, practicability, and repeatability. The cracking tolerance of an asphalt sample depends largely on some parameters which include the aggregate gradation used for the mix, air-voids and if additive is used, the type of additive also affects the CT-Index of the mix. The CT-Index of a sample can be calculated using equation 2-7 [41].

$$\mathbf{CT_{Index}} = \frac{\mathbf{t}}{\mathbf{62}} * \frac{\mathbf{G_f}}{|\mathbf{m_{75}}|} * \frac{\mathbf{l_{75}}}{\mathbf{D}} \qquad [2\text{-}7]$$

where,

CT_{Index} = cracking tolerance index

G_f = fracture energy, joules/m^2

$|m_{75}|$ = absolute value of the post-peak slope, N/m

l_{75} = displacement at 75% of post-peak slope, mm

D = specimen diameter, mm t = thickness of

specimen, mm

2.7.3 Marshall Stability and Flow

Marshall stability and flow test is used to evaluate the performance of the asphalt mixture. It is well known that the Marshall stability and flow test is the most common test used for the development of an asphalt mixtures design based on main parameters which include stability, flow, density, and air voids. The significant benefit for the application of Marshall mix design for asphalt mixtures is that the method provides adequate attention to the density and void properties, and these parameters ensure good volumetric ratios for the asphalt mixes. An advantage of this method is that the test setup is simple, the equipment for the test is portable and not expensive which makes it easy for remote quality control operations.

Marshall stability and flow test can be conducted in accordance with ASTM D6927-15 [42] specification using compacted specimens of 100mm in diameter and 60mm in height. Prior to the test the samples are prepared and compacted using a Marshall hammer. Marshall test is conducted using a load-deformation recorder together with a load cell and an automatic recording device which recorded both the stability and flow values of a sample. During the test, the maximum resistance load obtained during a constant rate of the deformation loading sequence is defined as the Marshall stability value of the sample. Marshall flow for the sample is defined as the maximum amount of deformation at the point of failure of the sample.

Chapter 3 Materials

Three materials were used in this study for preparation of mixtures. Along with two different types of aggregates, the two TSRU tailings samples and one bitumen froth sample were used in this study. The detail properties and characteristics of the samples are explained in this chapter.

3.1. TSRU Tailings

TSRU tailings composition differs depending on TSRU treatment extents and collection points in oil-sand industries. The variation of solvent and solid content can highly impact the characteristics of the TSRU tailings. In this research, two samples of TSRU materials were used (with and without water). The one that contained water was called wet TSRU and the dry one was called dry TSRU. After oven-drying both the samples, it was observed that the color of wet TSRU was black and the dry one was grey. From the visual appearances, dry TSRU looked like sandy and wet TSRU looked like dark like asphaltenes indicating there is might be more organic content or asphaltenes in wet TSRU. Figures 3-1 presents the TSRU samples in oven-dry conditions.

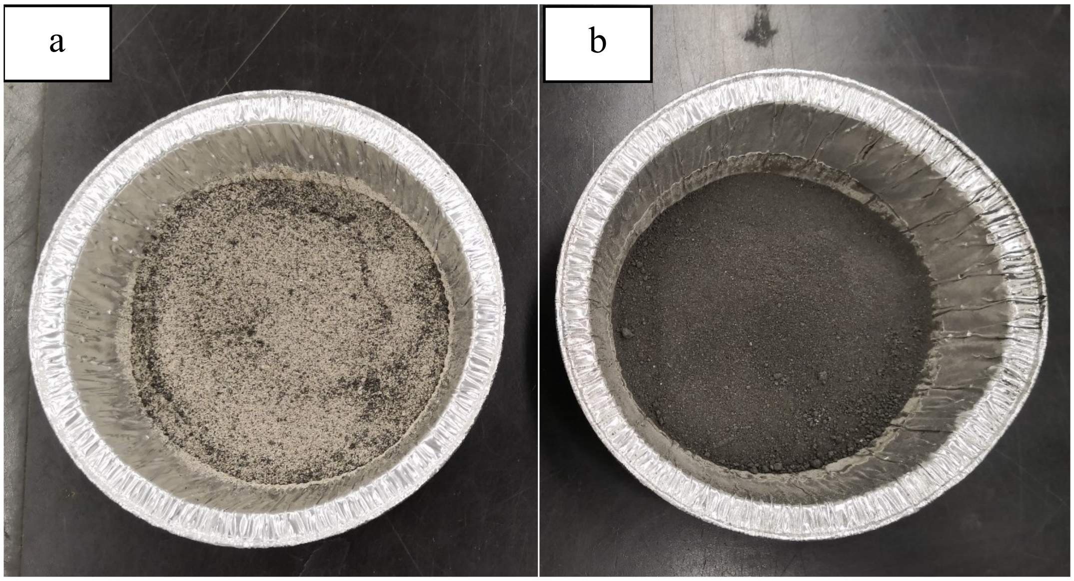

Figure 3-1 a. TSRU dry; b. TSRU wet in oven-dry condition

There was not much study where the physical properties such as specific gravity, grain size distribution, clay content, organic content of TSRU tailings were discussed. Hence, different characteristics of both TSRU tailing samples were investigated by different laboratory tests to understand the physical properties of TSRU tailings. Some of the important characteristics of TSRU tailings are enlisted here.

3.1.1 Specific Gravity

By conducting the specific gravity test as per ASTM D854-17 [43] after oven-drying the samples at 110 °C for 24 hr, the specific gravities of dry and wet TSRU tailings were calculated as per the following equation:

$$G_s = \frac{\alpha . Ms}{Ms + M1 - M2} \qquad [3\text{-}1]$$

where,

G_s = specific gravity

M_s= weight of dry soil (g)

M_1= weight of pycnometer + water at T_1 °C

M_2= weight of pycnometer + water + sample at T_2 °C

α = ratio of density of water at T °C to 20°C, T= (T_1+ T_2) °C

The specific gravities of dry and wet TSRU tailings were found to be 2.64 and 1.78, respectively. In order to evaluate the changes in physical properties of TSRU materials at higher temperatures, both TSRU samples were heated at 500 °C for 24 hr in an ignition oven in order to achieve a constant mass loss, as per ASTM D7348-21 [44]. The specific gravity was then calculated again. After ignition, the specific gravities of the dry and wet TSRU samples increased to 2.73 and 2.76, respectively. Figure 3-2 shows the temperature sensitivity of specific gravity for both type of TSRU tailings. Generally, for sandy soil, the specific gravity remains in the range of 2.65-2.67, whereas for silty sand soil the range is 2.67-2.70 and for inorganic clay it is 2.70-2.80 [45]. The obtained results showed that after ignition, the specific gravity of both types of TSRUs increased. Though the change is not that high for TSRU dry but for TSRU wet the change is quite significant. The possible reason behind that could be the organic content of TSRU wet. According to visual observation and specific gravity values, it could be concluded that the material is like silty sand soil. Dry TSRU physical property seems to change significantly with temperature variation while wet TSRU physical property does not.

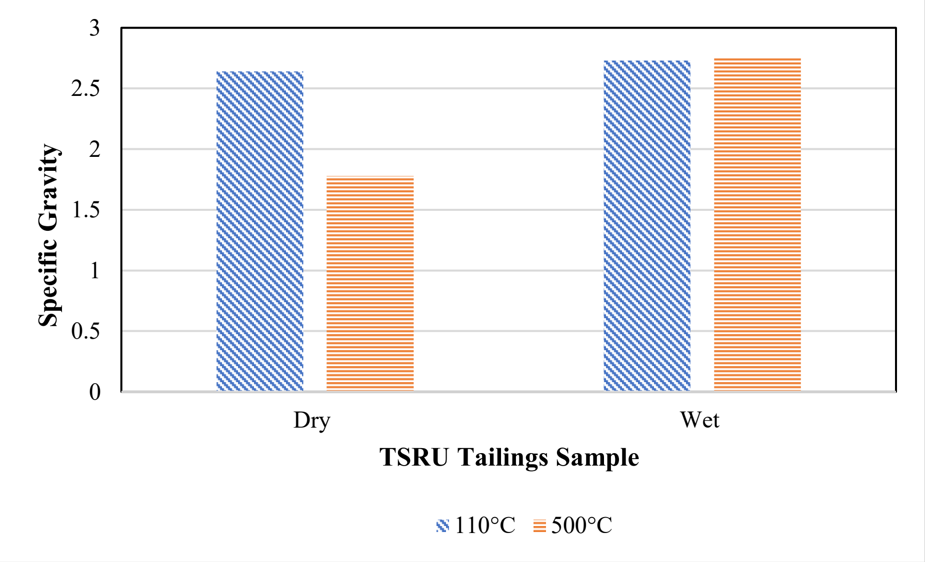

Figure 3-2 Comparison of specific gravity of TSRU tailings samples

3.1.2 Grain Size Distribution

The gradations of both TSRU samples were determined through sieve analysis and hydrometer analysis, keeping the samples at 500 °C for 24 hr (ignition method). The particles coarser than no. 200 sieve was graded using sieve analysis [46] and the fine particles (passing through no. 200 sieve) was graded with hydrometer analysis [47]. For the gradations of both TSRU tailings types are shown in Figure 3-3. The figure shows that wet TSRU tailings is slightly finer than dry TSRU material.

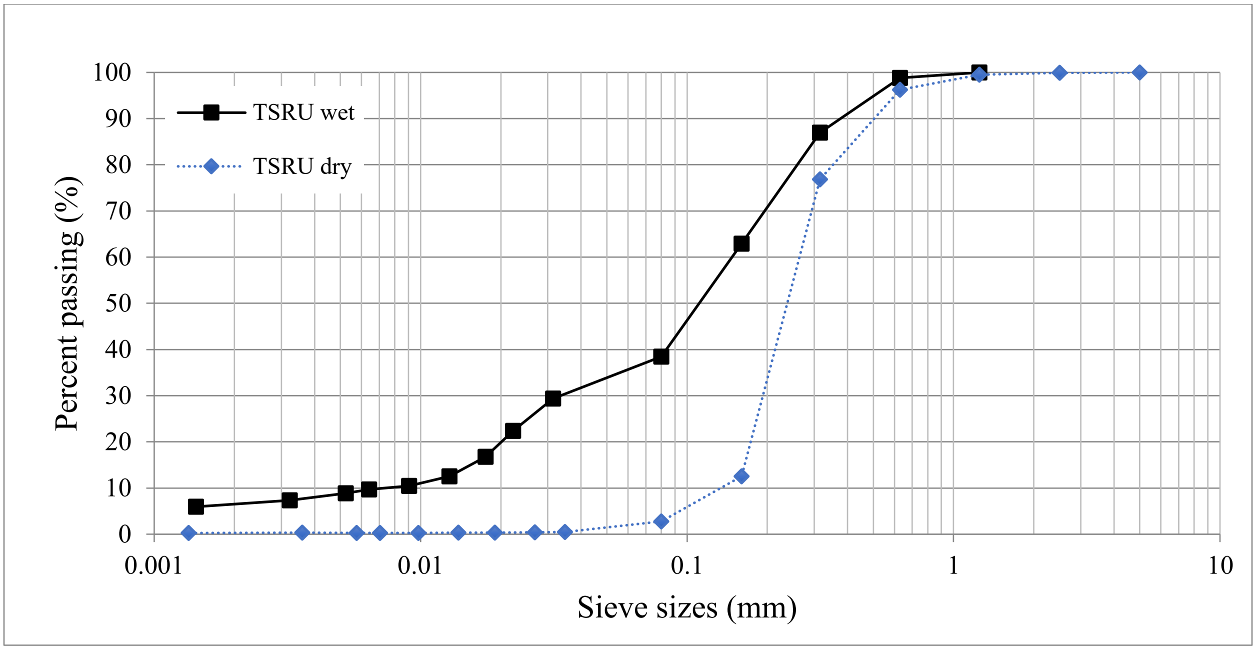

Figure 3-3 Comparisons of gradations of TSRU tailings samples.

3.1.3 SARA Analysis

The saturate, aromatic, resin and asphaltenes (SARA) test was conducted using the clay-gel adsorption chromatography method following ASTM D2007-20 [48]. The results are shown in Table 3-1. It was found that dry TSRU material is composed of 2% organic and 98% solid content, while wet TSRU, after removing the percentage of water, is composed of 40% organic and 60% solid content. It was also observed that the total saturate, aromatic, resin, and asphaltenes content did not add up to exactly 100% of the non-solid content in the samples as expected. This could be attributable to the presence of inorganic salts in the sample. Organic solvents were used for this test and the solubility of these inorganic salts in the organic solvents is very low. While the amount of inorganic salts was not significant, the process of completely removing the salts would have been time-consuming and expensive. A margin of error of 5%–10% was considered acceptable for the purpose of this experiment. Moreover, we were particularly interested in the asphaltenes content because it is used as a constituent material in pavement for roads. Based on the analysis, the organic content or asphaltenes of wet TSRU material was found to be higher than that of dry TSRU tailings, indicating that wet TSRU material is better in terms of binding properties for mix preparation, as per the literature [16].

Table 3-1 SARA analysis of TSRU tailings types.

TSRU Tailings	Saturates	Asphaltenes	Resins	Aromatics
Dry	12.83%	40.47%	24.83%	16.06%
Wet	10.58%	44.07%	21.90%	13.66%

3.1.4 Hydrocarbon Phase

The percentage of hydrocarbon phase (asphaltenes, molten, etc.) in both types of TSRU materials was calculated, following ASTM D6307-19 [49]. This standard, it should be noted, is typically used to calculate the asphalt content, but here it was used to calculate the total hydrocarbon phase as follows.

$$\text{\% Hydrocarbon} = \left(\frac{M_A - M_B}{M_A} \times 100\right) - C_F \qquad [3\text{-}2]$$

where

M_A= total mass of sample prior to ignition, gr

M_B= total mass of sample after ignition, gr

C_F= calibration factor ($C_F = 0$ in this case)

The hydrocarbon percentage of wet and dry TSRU materials were found to be 39.69% and 3.61%, respectively, again indicating that wet TSRU material is better than dry TSRU material for mixture preparation [17]. Thus, wet TSRU material was selected over its dry counterpart for the mix design.

3.1.5 Atterberg Limit

For further analysis of wet TSRU material, the Atterberg limit test was conducted for the wet TSRU sample following ASTM D4318-17 [50]. Liquid limit (LL), plastic limit (PL), and plasticity index (PI) values were found 42%, 39%, and 3% respectively. It should be noted that, generally, soils with a high PI tend to be clays, whereas soils with a lower PI value tend to have little clay content. Moreover, if the PI is 0, the soil is considered non-plastic, if PI < 7, the soil is slightly plastic, if it is within the range of 7–17, it is medium plastic, and, if PI > 17, then it is considered to be highly plastic [51]. The PI of the wet TSRU sample was found to be 3% (i.e., < 7), indicating that wet TSRU material is slightly plastic and implying that it is mainly composed of silt and has a very low clay content. A high PI, it should be noted, generally indicates a low shear strength, and also entails that more shrinkage will occur during drying. On the other hand, a low PI indicates that the soil will change significantly in consistency even with a small change in water content [52]. Accordingly, given its low plasticity, wet TSRU material is not expected to be susceptible to significant shrinkage, although it may be highly sensitive to moisture

3.1.6 Methylene Blue Index (MBI)

The MBI test provides an indication of clay activity of the soil. It measures how much methylene blue (MB) dye can be absorbed by the sample as determined by a titration test. It is a measure of surface area as the MB effectively covers the clay surface. MBI is the milliequivalents of MB per 100g of sample. The methylene blue index (MBI) test, which gives an indication of the clay activity of soil, was conducted on the wet TSRU sample following ASTM C837-19 [53]. It is calculated by multiplying the MB volume (mls to titrate 100 g of sample) by normality of MB used. The MBI value of the wet TSRU sample was calculated according to following equation:

$$\text{MBI (meq / 100 g)} = \frac{\text{mls in MB} \times \text{Normality of MB}}{\text{gr of samples}} \times 100 \qquad [3\text{-}3]$$

The average MBI of three replicates of the wet TSRU material was found to be 1.73. For oil sands clay, it should be noted, the expected MBI of a pure sample is 14 [54], meaning that the MBI of the wet TSRU material under investigation in our study was comparatively low. Moreover, the clay content in the wet TSRU sample was calculated using following equation:

$$\%\ \text{clay} = [\text{MBI Meq} / 100\ \text{g} + 0.04] / 0.14 \qquad [3\text{-}4]$$

% clay was found to be 12.6%, indicating like the Atterberg limit test described above that the wet TSRU sample may be sensitive to moisture.

3.2 Bitumen Froth

One bitumen froth sample was used in the study. Figure 3-4 shows the bitumen froth sample used in the study.

Figure 3-4 Bitumen froth

3.2.1 SARA Analysis

The SARA test revealed that the BF used in this study is composed of 16.54% saturates, 22.67% asphaltenes, 32.84% resins, and 23.76% aromatics. The relatively high asphaltenes content of the BF sample points to its suitability for use as a binder in the mix design.

3.2.2 Viscosity

In accordance with AASHTO T 316-19 [55], a Brookfield rotational viscometer was used to measure the viscosity of the BF binder at different temperatures, with the results shown in Table 3-2.

Table 3-2 Viscosity of the BF at different temperatures.

Temperature (°C)	Viscosity (Pa.s)
70	3.48
80	1.72
90	0.95
100	0.57

The viscosity of asphalt binders can be used to determine the mixing and compaction temperatures of asphalt mixtures. According to the Federal highway administration (FHWA), compacting asphalt mixtures requires mixing and compaction under equiviscous temperature conditions corresponding to 0.170 ± 0.020 Pa.s for mixing and 0.280 ± 0.030 Pa.s for compaction [56]. It is worth mentioning that, because the BF sample contained water and the boiling point of water is 100 °C, the test temperature could not be raised to more than 100 °C. Although, by extrapolating the values, the ideal mixing temperatures were found to be more than 100 °C, in light of the test temperature limitation noted above, 95 °C was selected as the mixing temperature based on engineering judgment.

3.2.3 Rheological Property

To characterize the high-temperature properties of the BF, a rheology test was conducted on both unaged and aged BF samples. A dynamic shear rheometer (DSR) was used for the rheology test, which was conducted in accordance with AASHTO T 315-20 [57]. In this test, a spindle with a diameter of 25 mm and a 1 mm gap was used for the unaged samples. To simulate asphalt binder aging in the plant, the asphalt samples were aged using a rolling thin-film oven (RTFO) in accordance with AASHTO T240-21 [58]. Moreover, the temperatures at which the parameter $\boldsymbol{G^*/\sin\delta}$ dropped below 1.0 kPa and 2.2 kPa were recorded as the failure temperatures for the unaged and RTFO-aged binders, respectively. The lowest temperature reached between these two, meanwhile, was deemed to be the final continuous high-performance grade (PG) of the binder. Accordingly, the high-PG for the unaged binder was found to be 52, whereas, for the RTFO-aged binder, the high-PG was found to be 56.7, so the continuous PG grade test result was 52. This result indicates that the BF sample investigated is a suitable binder for use in pavement construction in cold-climate regions such as Alberta, Canada.

3.3 Granular Materials

The in-pit haul roads are constructed in order to support the loads from a fully loaded 797F haul truck. The operating life of these roads are usually 2 years (semi-permanent) or 5 years (permanent). During the construction of base and surface layer, limestone (75mm) and alluvial (40mm/75mm) are used respectively [59]. Subbase materials of this in-haul roads are sand or peak ground acceleration (Pga) till. As these materials have similar resilient modulus and friction angles, the materials are interchangeable in the design. The intent of providing pavement cross sections with two options for subbase material is to give operations flexibility to construct the cross section that matches the available site produced construction material and subgrade conditions. During construction, it is common for a single haul road to be constructed on multiple types of subgrades, such as ore grade oil sand, undisturbed clays, clay fills, or Pga till/sand. Usually, the thickness of alluvial layer is 0-0.6 m where the thickness of limestone layer is 0.6-1.5m [59] which is really high. In this research, limestone and alluvial are used as granular materials for base course stabilization with TSRU tailings with an aim of reducing the thickness of haul roads without any additional costs.

Figure 3-5 Granular materials (limestone and alluvial)

3.3.1 Limestone

75mm limestone is used in the base layer at in-haul roads. The base layer provides the main source of stiffness and bearing capacity to the road. The material needs to have high strength and stiffness to bear the loads and minimize deflection and rolling resistance. The material must be durable in order to not break down under repeated heavy loads and weathering. Crushed limestone can provide these properties, and was selected as the material for the base layer. The unit weight of this material is 21 kN/m^3. A friction angle of 42° was selected for this type of granular material. Resilient modulus values indicate that using a 75mm crushed limestone resilient modulus of 283 MPa at a depth range 0.6 to 1.5m is sufficient for haul road design. The value is considered to be representative of a 75mm crushed limestone compacted to 98% [59].

3.3.2 Alluvial

75mm or 40mm crushed alluvial gravel is the second granular material used in the study. It is used as a surface material for the haul roads. The main functions of the surface layer, or wearing course, is to provide traction to the haul trucks, be able to withstand all weather conditions and be strong to bear the haul truck load and provide minimal rolling resistance. The material must be durable in order to not break down under repeated heavy loads, and minimize dust creation. The surface also needs to be maintained with the equipment available on site such as dozers / graders. Properly specified crushed alluvial can provide these properties to the haul road surface. The unit weight of 40mm crushed gravel is 21 kN/m^3, while the unit weight of 75mm crushed gravel is 22.5 kN/m^3. A friction angle of 42° for both materials was adopted for the bearing capacity analysis. Resilient modulus values for 75 / 40mm crushed alluvial gravel recommended using a 75 / 40 mm crushed alluvial gravel resilient modulus of 594 MPa at a depth range of 0 to 0.6m based on the study results. The value is considered to be representative of a 75/40 mm crushed alluvial gravel compacted to 100% [59].

3.3.3 Physical Properties of Granular Materials

From the literature, some important physical properties of the both granular materials such as grain size distribution, strength, Atterberg limits, optimum moisture content etc were not listed in any previous literature. Thus, these properties are investigated by some laboratory tests.

3.3.3.1 Atterberg Limits

The atterberg limit test was conducted following ASTM D4318-17 [50]. Limestone was non-plastic because the particles did not mix with water as. For alluvial, the LL, PL, and PI were found 33%, 23%, and 10%, respectively. Normally, soils with PI within 7-17 are considered to be medium plastic and for silty clay of medium plasticity, the LL is 27-55 % and the PL is 12-33% [60]. Thus, alluvial (sample 2) has medium plasticity

3.3.3.2 Modified Proctor Test

The modified proctor test was conducted for both aggregate samples following ASTM D1557-10 [61] to determine the maximum dry density (MDD) and OMC. In case of alluvial, the MDD and OMC were found as 22.2 kN/m^3 and 6.0% respectively which were close to the values obtained for limestone (MDD= 22.0 kN/m^3 and OMC= 5.8%).

3.3.3.3 California Bearing Ratio Test

California bearing ratio test is a good indicator of the strength of the granular materials. In order to measure the strength of the aggregates in wet conditions, soaked CBR test was conducted on both aggregate samples according to ASTM D1883-21 [62]. This test gives an idea of the strength of a material using a standardized penetration test. The soaked CBR test results showed that limestone had a CBR value of 43% which was lower than the CBR value obtained for alluvial (that is 52.5%). The CBR values indicate there is no significant difference between the aggregates in terms of strength. Thus, both types of granular materials were used in the mix design process.

3.3.3.4 Grain Size Distribution

The gradations of the granular materials were determined by wet sieving following ASTM D1140-17 [63]. Table 2-3 shows the grain size distribution of both limestone and alluvial. The result shows that there is not a significant difference between the gradations of aggregate materials but the gravel alluvial is finer than limestone.

Table 3-3 Grain size distribution of granular materials.

Limestone		Alluvial	
Size (mm)	**Passing (%)**	**Size (mm)**	**Passing (%)**
90.00	100.00	90.00	100.00
75.00	94.97	75.00	94.98
50.00	94.97	50.00	94.98
25.00	84.62	25.00	71.83
12.50	68.58	12.50	56.53
5.00	54.22	5.00	37.48
1.25	33.01	1.25	25.00
0.63	24.82	0.63	22.18
0.32	19.98	0.32	20.53
0.16	15.97	0.16	19.36
0.08	11.46	0.08	18.65

Chapter 4 Performance Evaluation of Stabilized Base Courses Comprising TSRU Materials and Bitumen Froth Derived from Alberta Oil Sands

4.1 Abstract

Tailings solvent recovery unit materials and bitumen froth are byproducts derived from Alberta oil sands. This study investigates the application of these byproducts in improving granular base course mechanical properties. In this research, the properties of two tailings solvent recovery unit samples, one bitumen froth sample, and two aggregates are evaluated using various laboratory tests. The tailings solvent recovery unit-modified mixtures are prepared at different concentrations of bitumen froth, and their properties are investigated using Indirect Tensile Strength and Marshall Stability tests. The results show that tailings solvent recovery unit modification results in a higher tensile strength. The granular layer thickness can be reduced by 42% and 60% for modified samples made with both aggregates. It should be noted that although the modified samples are found to satisfy the unsoaked indirect tensile strength requirement, they fail to meet the soaked indirect tensile strength requirement.

4.2 Introduction

Innovative solutions such as stabilization of the base course using various stabilizing agents have become popular in recent years as means of enhancing the properties of the unbound layer [12]. Portland cements or asphalt emulsions can be used for this purpose. However, the use of Portland cement in stabilized mixtures has several drawbacks in comparison to other stabilizing agents, such as high cost and increased risk of thermal cracking. In addition, it is not environment-friendly because a significant volume of CO_2 is released into the atmosphere during cement production [13]. Asphalt emulsion, meanwhile, also has a high cost, and it needs to cure for a considerable amount of time before it can achieve the required binding properties [11], resulting in lower mechanical strength at the early stages of the pavement's service life [15]. Given these economic constraints and environmental concerns, researchers are continually seeking alternative and creative materials.

Tailings Solvent Recovery Unit or TSRU tailings (or, materials obtained from oil sands bitumen production) are waste materials with no notable use in current practice. TSRU tailings are a compelling and cost-effective material to consider for use in pavement construction. In froth treatment plants, a paraffinic solvent is added to the froth to help separate bitumen from water and solids. The tailings containing water and solids are sent to the TSRU from the froth treatment plant in order to recover the paraffinic solvent. Tailings that are processed by the TSRU, it should be noted, are referred to as TSRU tailings. The TSRU tailings stream mainly contains water, asphaltenes, fines, solids, bitumen, and residual solvent. The solvent content in the tailings can vary between 5% and 10%, with the exact composition of the tailings stream varying according to operating conditions [16]. TSRU tailings have higher pyrite and residual asphaltenes content compared to conventional naptha-treated froth treatment tailings [16]. Disposal and management of TSRU tailings is a significant concern for the oil sands industry. TSRU tailings are typically disposed of in tailings ponds. The Alberta Energy Regulator (AER) established a tailings management directive (Directive 085) and proposed an outcome- and risk-based approach to hold operators accountable for managing their fluid tailings [64]. In this context, using TSRU tailings for base course stabilization could offer economic and environmental benefits.

Bitumen Froth is another byproduct of bitumen production from oil sands. Froth treatment is an important step in the process of recovering bitumen from oil sands via surface mining. Typically, the recovered BF consists of 60 wt% bitumen, 30 wt% water, and 10 wt% mineral solids [19]. In froth treatment, water droplets and mineral solids are separated from the organic bitumen product using hydrocarbon solvents, although the micrometer-sized mineral particles (mainly clays) and water-in-oil emulsion droplets present in BF are difficult to remove [20]. These fine mineral particles are the main detriments in stabilizing the water-in-oil emulsions, where emulsified water droplets are easy to destabilize and remove in the absence of fine mineral particles [19,20]. The treated BF can still contain, on average, 2 wt% to 5 wt% water and 0.5 wt% to 1 wt% solids [22,23].

In this study, the impact of TSRU modification in stabilized asphalt mixtures and the change in the granular layer thickness after TSRU modification is investigated. From previous studies, it is expected that TSRU modification will improve the properties of stabilized asphalt mixtures due to the presence of asphaltenes. Asphaltenes, it should be noted, can be obtained from various sources, including oil sands, crude oil, asphaltite, tar sand, and bituminous coal [65]. The application of asphaltenes can improve the rheological properties of asphalt mixtures [66,67]. Kamran et al. [17] found that mixtures stabilized with an asphalt emulsion exhibit significantly improved high-temperature properties in comparison with unmodified mixtures. Basavarajappa et al. [18], meanwhile, concluded that the addition of asphaltenes to asphalt emulsion increases the shear modulus of the asphalt emulsion base binder and, as a result, improves the stiffness and rutting resistance of the mixture. In our research, BF is used in place of asphalt emulsion. The findings of previous studies indicated that the addition of TSRU materials to BF may improve the properties of stabilized asphalt mixtures because of the presence of asphaltenes in TSRU materials.

4.3 Objectives

The main objective of our study was to investigate the impact of TSRU tailings, a waste material of the oil sands industry, in enhancing the performance properties of stabilized mixes using granular materials and BF, thereby enabling a reduction in the required thickness of the granular layer (and leading to material/cost savings and environmental benefits). To achieve this goal, first the characteristics of TSRU materials, BF, and granular material were evaluated using a number of laboratory testing methods. Moreover, several laboratory tests were conducted to determine which TSRU sample would perform best in the mixture and to investigate the impact of adding it to the granular materials and BF. TSRU-modified mixtures were prepared at different concentrations of BF. The mechanical properties of all the mixtures were investigated by conducting an ITS test and a Marshall Stability test. The impact of TSRU materials on tensile strength and the change in the granular layer thickness after modification with TSRU materials was also investigated.

4.4 Materials

4.4.1 Aggregates

Two types of aggregate materials, known as limestone (Sample 1) and gravel alluvial (Sample 2), were used in this study. These aggregate materials are commonly used in the construction of mine haul roads [59]. Both of the granular materials were provided by an oil sand operator that operates in the Alberta oil sands region. Laboratory tests were conducted to evaluate the physical properties of both aggregate types.

4.4.1.1 Atterberg Limit of Aggregates

The Atterberg limit test was conducted following ASTM D4318-17 [50]. In the case of Sample 1, the particles did not mix with water as they are non-plastic. For Sample 2, the Liquid Limit or LL, Plastic Limit or PL, and Plasticity Index or PI were found to be 33%, 23%, and 10%, respectively. Normally, soils with a PI within the range 7–17 are considered to be of medium plasticity, and a silty clay of medium plasticity has been reported in the literature to have an LL within the range 27%–55% and a PL within the range 12%–33% [60]. Accordingly, the gravel alluvial (Sample 2) was determined to be of medium plasticity.

4.4.1.2 Modified Proctor Test

A modified proctor test was conducted for both aggregate samples following ASTM D1557-10 [61] to determine the maximum dry density or MDD and optimum moisture content or OMC. In the case of Sample 2, the MDD and OMC were found to be 22.2 kN/m^3 and 6.0%, respectively—close to the values obtained for Sample 1 (MDD = 22.0 kN/m^3 and OMC = 5.8%).

4.4.1.3 California Bearing Ratio Test

In order to measure the strength of the aggregates in wet conditions, soaked California bearing ratio test was conducted on both aggregate samples according to ASTM D1883-21 [62]. Based on this test, which ascertains the strength of a material using standardized penetration, Sample 1 was found to have a CBR value of 43%-lower than the CBR value obtained for Sample 2 (i.e., 52.5%). According to roads and airfields, department of army technical manual [68], for high quality base course, the minimum CBR value should be 80%. As per IRC method of design of flexible pavements [59] for sub-base course, the minimum CBR value is 20% for 2 million standard axle (MSA) and 30% for traffic exceeding 2 MSA. From the CBR values of the aggregates, it could be said that these can be used as high quality sub-base materials. This indicates that there is no significant difference between the two aggregates in terms of strength. Thus, both types of granular materials were used in the mix design process.

4.4.1.4 Grain Size Distribution of Aggregates

The gradations of the granular materials were determined using a wet sieving procedure following ASTM D1140-17 [63], with the results provided in Table 4-1. Table 4-1 shows that there is not a significant difference in gradation between the aggregate materials, but that the gravel alluvial (Gradation 2) is finer than the limestone (Gradation 1).

Table 4-1 Grain size distribution of granular materials.

Gradation 1		Gradation 2	
Size (mm)	**Passing (%)**	**Size (mm)**	**Passing (%)**
90.00	100.00	90.00	100.00
75.00	94.97	75.00	94.98
50.00	94.97	50.00	94.98
25.00	84.62	25.00	71.83
12.50	68.58	12.50	56.53
5.00	54.22	5.00	37.48
1.25	33.01	1.25	25.00
0.63	24.82	0.63	22.18
0.32	19.98	0.32	20.53
0.16	15.97	0.16	19.36
0.08	11.46	0.08	18.65

4.4.2 TSRU Tailings

Two types of TSRU tailings samples were used in this study, these samples having been provided by an oil sand operator, an oil and gas company operating in the Alberta oil sands. Both wet and dry TSRU samples were provided, as shown in Figure 4-1. A series of tests were conducted to determine the physical properties of both TSRU materials and thereby determine which one is better suited for use in the mix design.

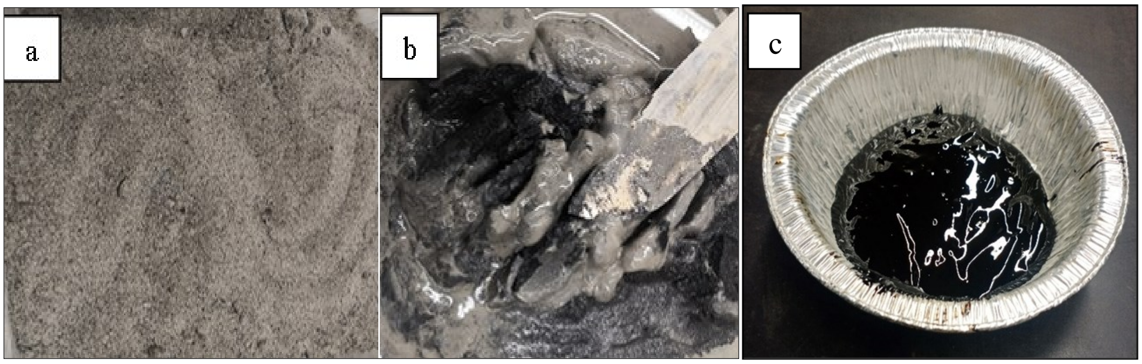

Figure 4-1 (a) dry TSRU material; (b) wet TSRU material; (c) Bitumen froth.

4.4.2.1 Specific Gravity

The specific gravity test was conducted as per ASTM D854-17 [45] after oven-drying the samples at 110 °C for 24 hr. The specific gravities of dry and wet TSRU materials were found to be 2.64 and 1.78, respectively. In order to evaluate the changes in physical properties of TSRU materials at higher temperatures, both TSRU samples were heated at 500 °C for 24 hr in an ignition oven in order to achieve a constant mass loss, as per ASTM D7348-21 [46]. The specific gravity was then calculated again. After ignition, the specific gravities of the dry and wet TSRU samples increased to 2.73 and 2.76, respectively.

4.4.2.2 Grain Size Distribution of TSRU Tailings

The gradations of both TSRU samples were determined through sieve analysis and hydrometer analysis, keeping the samples at 500 °C for 24 hr (ignition method). For the purpose of comparison, the gradations are shown in Figure 3-2. The results show that wet TSRU material is slightly finer than dry TSRU material.

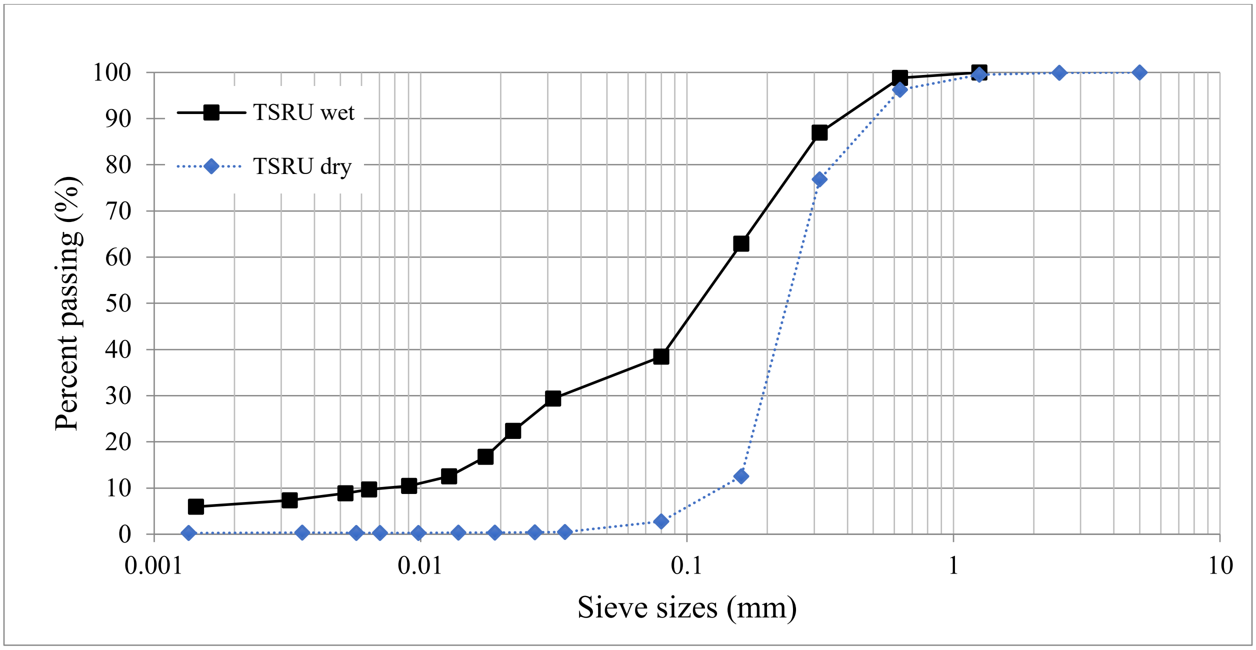

Figure 4-2 Comparisons of gradations of TSRU samples.

4.4.2.3 SARA Analysis of TSRU Tailings

The saturate, aromatic, resin and asphaltenes or SARA test was conducted using the clay-gel adsorption chromatography method following ASTM D2007-20 [48]. The results are shown in Figure 4-3. It was found that dry TSRU material is composed of 2% organic and 98% solid content, while wet TSRU, after removing the percentage of water, is composed of 40% organic and 60% solid content. It was also observed that the total saturate, aromatic, resin, and asphaltenes content did not add up to exactly 100% of the non-solid content in the samples as expected. This could be attributable to the presence of inorganic salts in the sample. Organic solvents were used for this test and the solubility of these inorganic salts in the organic solvents is very low. While the amount of inorganic salts was not significant, the process of completely removing the salts would have been time-consuming and expensive. A margin of error of 5%–10% was considered acceptable for the purpose of this experiment. Moreover, we were particularly interested in the asphaltenes content because it is used as a constituent material in pavement for roads. Based on the analysis, the organic content of wet TSRU material was found to be higher than that of dry TSRU material, indicating that wet TSRU material is better in terms of binding properties for mix preparation, as per the literature [17].

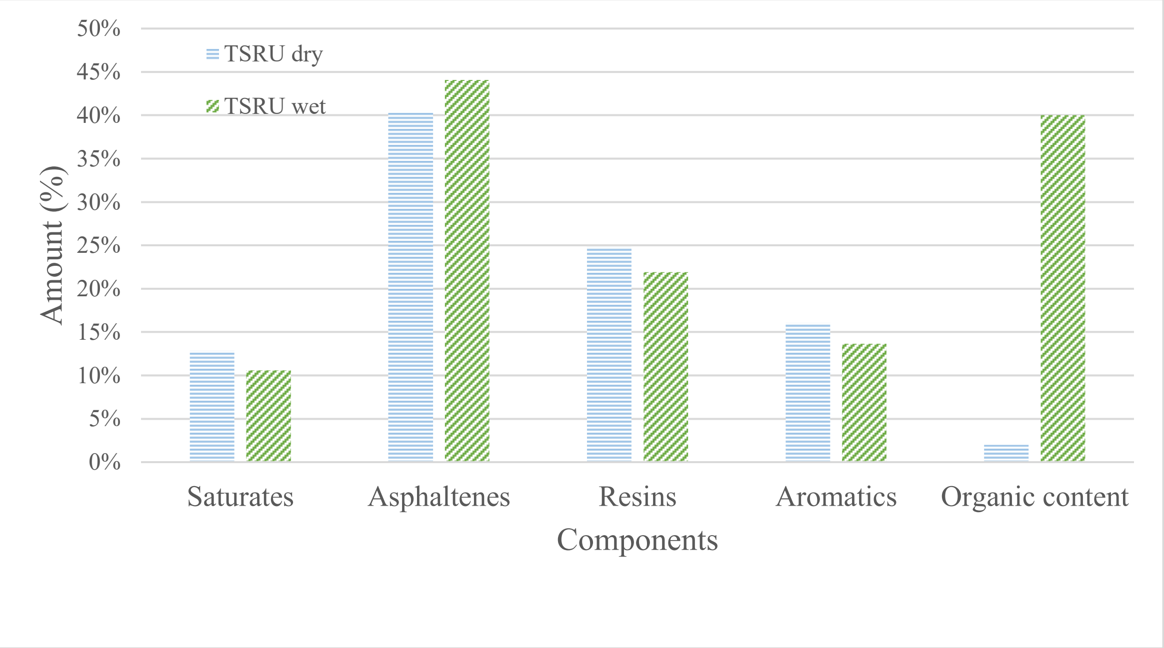

Figure 4-3 SARA analysis comparison for TSRU samples.

4.4.2.4 Hydrocarbon Phase

The percentage of hydrocarbon phase (asphaltenes, molten, etc.) in both types of TSRU materials was calculated, following ASTM D6307-19 [49], using equation 4-1. This standard, it should be noted, is typically used to calculate the asphalt content, but here it was used to calculate the total hydrocarbon phase as follows.

$$\% \text{ Hydrocarbon} = \left(\frac{M_A - M_B}{M_A} \times 100\right) - C_F \quad [4\text{-}1]$$

where

M_A= total mass of sample prior to ignition, gr

M_B= total mass of sample after ignition, gr

C_F= calibration factor (C_F = 0 in this case)

The hydrocarbon percentage of wet and dry TSRU materials were found to be 39.69% and 3.61%, respectively, again indicating that wet TSRU material is better than dry TSRU material for mixture preparation [17]. Thus, wet TSRU material was selected over its dry counterpart for the mix design.

4.4.2.5 Atterberg Limit of TSRU Tailings

For further analysis of wet TSRU material, the Atterberg limit test was conducted for the wet TSRU sample following ASTM D4318-17 [50]. LL, PL, and PI values were found 42%, 39%, and 3% respectively. It should be noted that, generally, soils with a high PI tend to be clays, whereas soils with a lower PI value tend to have little clay content. Moreover, if the PI is 0, the soil is considered non-plastic, if PI < 7, the soil is slightly plastic, if it is within the range of 7–17, it is medium plastic, and, if PI > 17, then it is considered to be highly plastic [51]. The PI of the wet TSRU sample was found to be 3% (i.e., < 7), indicating that wet TSRU material is slightly plastic and implying that it is mainly composed of silt and has a very low clay content. A high PI, it should be noted, generally indicates a low shear strength, and also entails that more shrinkage will occur during drying. On the other hand, a low PI indicates that the soil will change significantly in consistency even with a small change in water content [54]. Accordingly, given its low plasticity, wet TSRU material is not expected to be susceptible to significant shrinkage, although it may be highly sensitive to moisture.

4.4.2.6 Methylene Blue Index (MBI)

The MBI test gives an indication of the clay activity of soil, was conducted on the wet TSRU sample following ASTM C837-19 [53]. The average MBI value of the wet TSRU sample was calculated accordingly using equation 4-2. The average MBI of three replicates of the wet TSRU material was found to be 1.73. For oil sands clay, it should be noted, the expected MBI of a pure sample is 14 [54], meaning that the MBI of the wet TSRU material under investigation in our study was comparatively low. Moreover, the clay content in the wet TSRU sample was calculated using equation 4-3 and was found to be 12.6%, indicating like the atterberg limit test described above that the wet TSRU sample may be sensitive to moisture.

$$\text{MBI (meq / 100 g)} = \frac{\text{mls in MB} \times \text{Normality of MB}}{\text{gr of samples}} \times 100 \tag{4-2}$$

$$\text{\% clay} = [\text{MBI Meq / 100 g} + 0.04] / 0.14 \tag{4-3}$$

4.4.3 Bitumen Froth

As noted above, BF is a byproduct of bitumen production from Alberta oil sands. This binder is very soft and it contains a small amount of water–oil emulsion. In our study, one type of BF sample was used for the mixture preparation, along with granular materials and wet TSRU material, the BF having been provided by an oil sand operator (Figure 3-1c).

4.4.3.1 SARA Analysis of Bitumen Froth

The SARA test revealed that the BF used in this study is composed of 16.54% saturates, 22.67% asphaltenes, 32.84% resins, and 23.76% aromatics. The relatively high asphaltenes content of the BF sample points to its suitability for use as a binder in the mix design.

4.4.3.2 Viscosity

In accordance with AASHTO T 316-19 [55], a Brookfield rotational viscometer was used to measure the viscosity of the BF binder at different temperatures, with the results shown in Table 4-2.

Table 4-2 Viscosity of the BF at different temperatures.

Temperature (°C)	Viscosity (Pa.s)
70	3.48
80	1.72
90	0.95
100	0.57

The viscosity of asphalt binders can be used to determine the mixing and compaction temperatures of asphalt mixtures. According to the FHWA [56], compacting asphalt mixtures requires mixing and compaction under equiviscous temperature conditions corresponding to 0.170 ± 0.020 Pa.s for mixing and 0.280 ± 0.030 Pa.s for compaction. It is worth mentioning that, because the BF sample contained water and the boiling point of water is 100 °C, the test temperature could not be raised to more than 100 °C. Although, by extrapolating the values, the ideal mixing temperatures were found to be more than 100 °C, in light of the test temperature limitation noted above, 95 °C was selected as the mixing temperature based on engineering judgment.

4.4.3.3 Rheology Test

To characterize the high-temperature properties of the BF, a rheology test was conducted on both unaged and aged BF samples. A DSR was used for the rheology test, which was conducted in accordance with AASHTO T 315-20 [57]. In this test, a spindle with a diameter of 25 mm and a 1 mm gap was used for the unaged samples. To simulate asphalt binder aging in the plant, the asphalt samples were aged using a RTFO in accordance with AASHTO T240-21 [58]. Moreover, in accordance with the specifications in AASHTO M320-21 [70] for testing 25 mm-diameter samples at high temperatures, the temperatures at which the parameter $\boldsymbol{G^*/\sin\delta}$ dropped below 1.0 kPa and 2.2 kPa were recorded as the failure temperatures for the unaged and RTFO-aged binders, respectively. The lowest temperature reached between these two, meanwhile, was deemed to be the final continuous high-performance grade of the binder. Accordingly, the high-PG for the unaged binder was found to be 52, whereas, for the RTFO-aged binder, the high-PG was found to be 56.7, so the continuous PG grade test result was 52. This result indicates that the BF sample investigated is a suitable binder for use in pavement construction in cold-climate regions such as Alberta, Canada.

4.5 Experimental Program

In a study, Kamran et al. [17] concluded that asphaltenes modification can improve significantly the high-temperature properties of modified mixtures. It was thus hypothesized that both the wet TSRU material and the BF would improve the high-temperature properties of the modified mixtures, since they have 44.07% and 22.67% asphaltenes content, respectively.

4.5.1 Mixture Preparation

In this study, two gradations, referred to as Gradation 1 and Gradation 2, were used for mixture preparation. These gradations were kept consistent for all subsequent mixtures. The OMCs of both aggregate types were calculated using the modified proctor test, and they were found to be 5.8% and 6.0% for gradations 1 and 2, respectively.

First, both aggregate samples were oven-dried at 110 °C for 24 hr and crushed using a wooden hammer. Then, only the particles passing through a 20-mm sieve were selected for the preparation of the mixtures. The mixing process was initiated by adding the aggregates and wet TSRU material at 95 °C to the blend to reach the desired OMC (i.e., 5.8% for Gradation 1 and 6% for Gradation 2). The total water content in the mixtures was considered the OMC of the aggregates. During the mixing process, no external water was added, but instead the moisture present in the wet TSRU material was used to achieve the OMC. BF that had been heated to 95 °C was then added to the mixture. (The BF was heated to 95 °C because of the water content, as explained above.) The samples were then mixed until the TSRU materials and BF were uniformly distributed throughout the aggregate blend. (The proportions of materials in the mix design are given in Table 4-3, while the modified gradations for aggregate Samples 1 and 2 are shown in Figure 4-4).

Table 4-3 Grain size distribution of granular materials.

Materials	**Used amount**		**Comment**
	Gradation 1	**Gradation 2**	
Total aggregates (g)	1,100	1,100	
BF (g)	44 (for 4% of total aggregates)	44 (for 4% of total aggregates)	Depends on percentage of BF
Required water for mix (g)	63.8	66	Based on optimum moisture content of aggregates
Moisture drop in TSRU (%)	20	20	Heated at 95 °C for 2 hr
Required TSRU (g)	224	217	Based on the required water
Asphaltenes in mix (%)	1.6	1.7	Only the powder asphaltenes in wet TSRU material (44.07%) is considered here—not the liquid asphaltenes in BF.

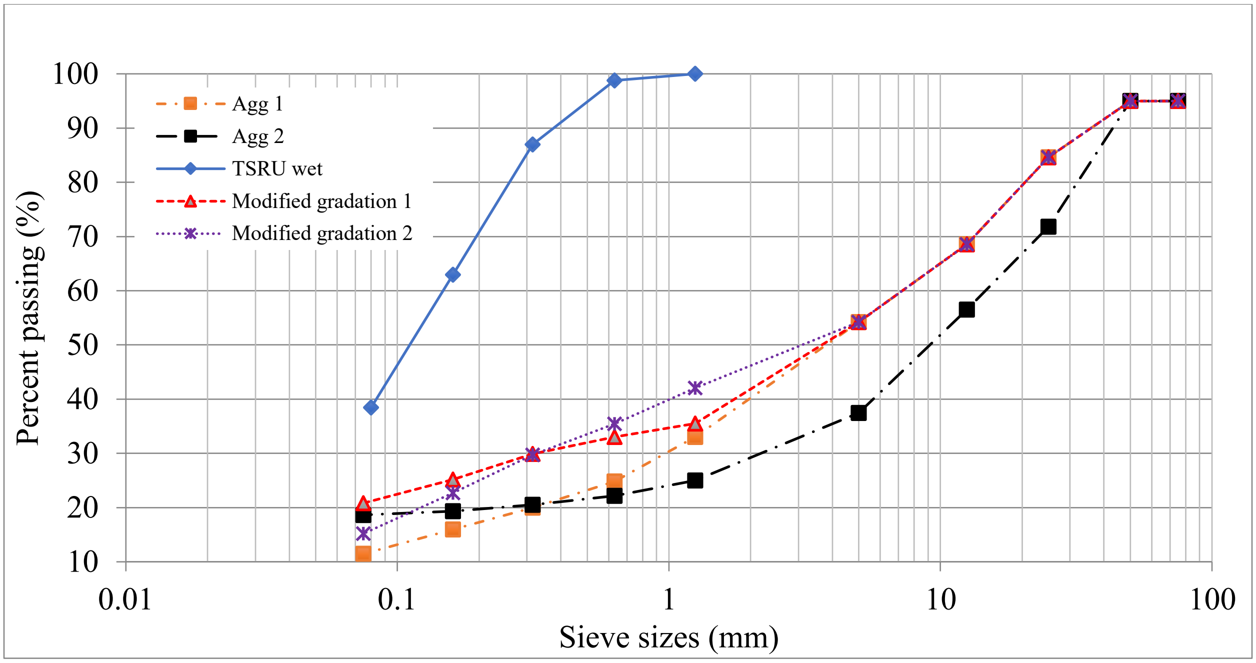

Figure 4-4 Modified gradation for aggregate Samples 1 and 2.

The samples were prepared by applying 75 blows with a Marshall hammer per side of the sample. For the unsoaked ITS test, three replicates were prepared for with BF contents of 0% to 6% (increasing in 1% intervals), while, for the soaked ITS test, three replicates with 4%, 5%, and 6% content were prepared. Finally, the samples were placed in an oven for curing at 40 °C for 72 hr, as specified in the TG2 guideline [71]. The cured samples were then kept at room temperature for at least 2 hr prior to extraction from the mould.

4.5.2 Indirect Tensile Strength

The unsoaked ITS test was performed according to AASHTO T 283-21 [38] for the samples containing BF content of 0% to 6% (increasing by 1% intervals) in order to determine the optimum BF. The soaked ITS test was then conducted on the saturated samples containing 4%, 5%, and 6% BF content. Prior to the soaked ITS test, the samples were conditioned in water at 25 °C for 24 hr. The ITS tests were conducted by applying loads at a rate of 50 mm/min. The maximum load applied to the sample until failure was recorded in order to calculate the tensile strength of the samples. (The ITS test setup and samples before and after cracking are shown in Figure 5.) The strength of each sample was calculated as per equation 4-4.

$$S_t = \frac{2000*P}{\pi*t*D} \qquad [4\text{-}4]$$

where

S_t = indirect tensile strength (kPa)

P = maximum applied load (N)

t = average height of specimen (mm)

D = diameter of specimen (mm)

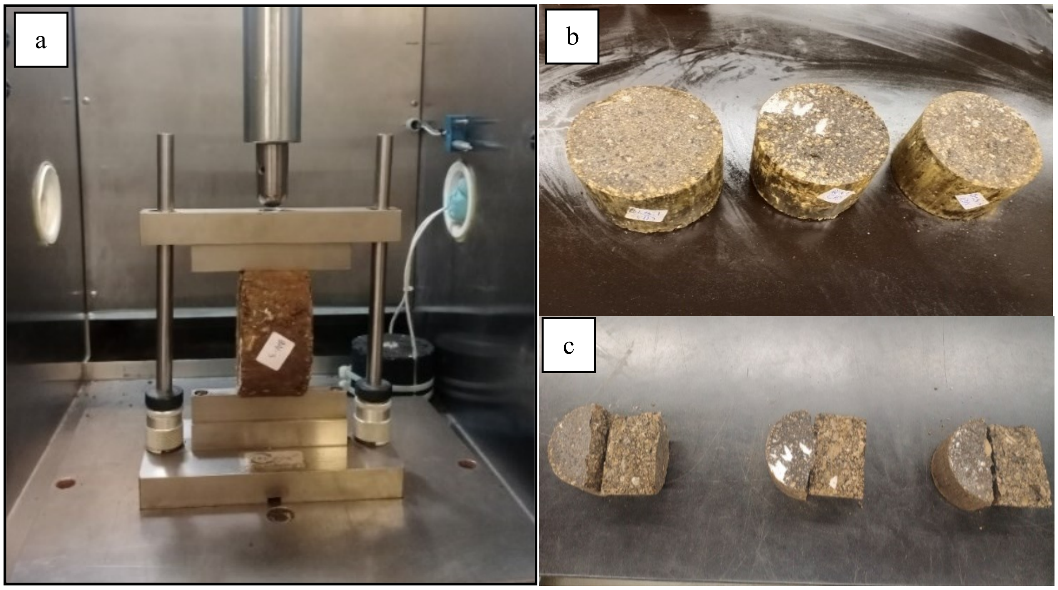

Figure 4-5 (a) ITS test set up; ITS samples (b) before and (c) after the test (gradation-1 samples with 3% BF).

The maximum soaked ITS value was observed at 5% BF for both gradations. Hence, 5.0% was deemed to be the optimum BF content for the mix design.

4.5.3 Impact of TSRU on Tensile Strength

In order to quantify the impact of TSRU modification on the mix design, mixtures were prepared for the optimum BF content (5%) but no TSRU material, and the results were compared to those of the samples with TSRU material and optimum BF.

4.5.4 Marshall Stability Test

To estimate the change in granular layer thickness after TSRU and BF modification, the CBR value of unmodified aggregates and the Marshall Stability value of the modified mixture needed to be obtained. For this purpose, the Marshall Stability test was conducted for both gradations with the optimum BF content in accordance with ASTM D6927-15 [42] using the same mix design process and compaction and curing procedures described above. Three replicates were prepared and conditioned for 30 minutes at 60 °C in a water bath prior to testing, as per the mentioned standard.

4.6 Results and Discussion

4.6.1 Indirect Tensile Strength Test

The unsoaked ITS test results for the TSRU-modified samples for both gradations are presented in Figure 4-6. The figure shows that the maximum unsoaked ITS value was achieved after adding 3.0% BF by weight to aggregate Gradation 1, whereas the optimum BF for Gradation 2 was 5.0%. The soaked ITS results, meanwhile, are presented in Figure 4-7. As shown in this figure, the maximum soaked ITS was observed at 5% BF content for both gradations. Hence, 5% BF was deemed to be the optimum BF content for this mix design, as noted above.

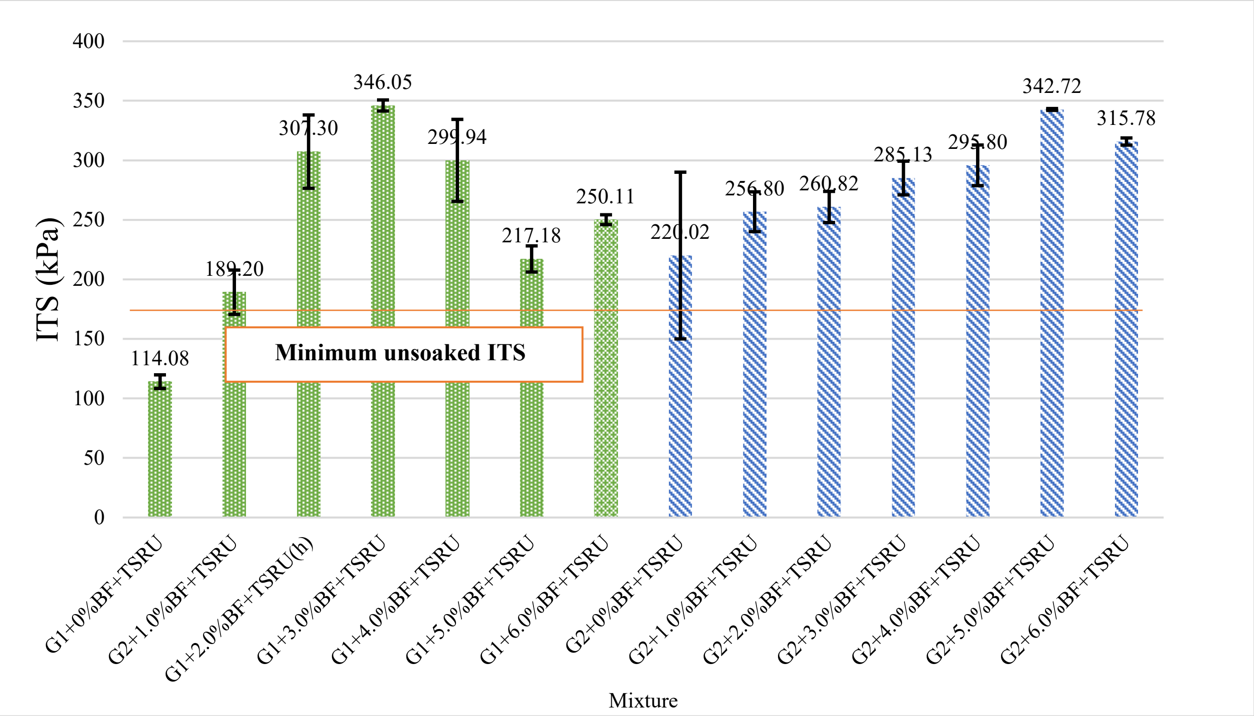

Figure 4-6 Unsoaked ITS test results for the TSRU-modified samples prepared with Gradations 1 and 2.

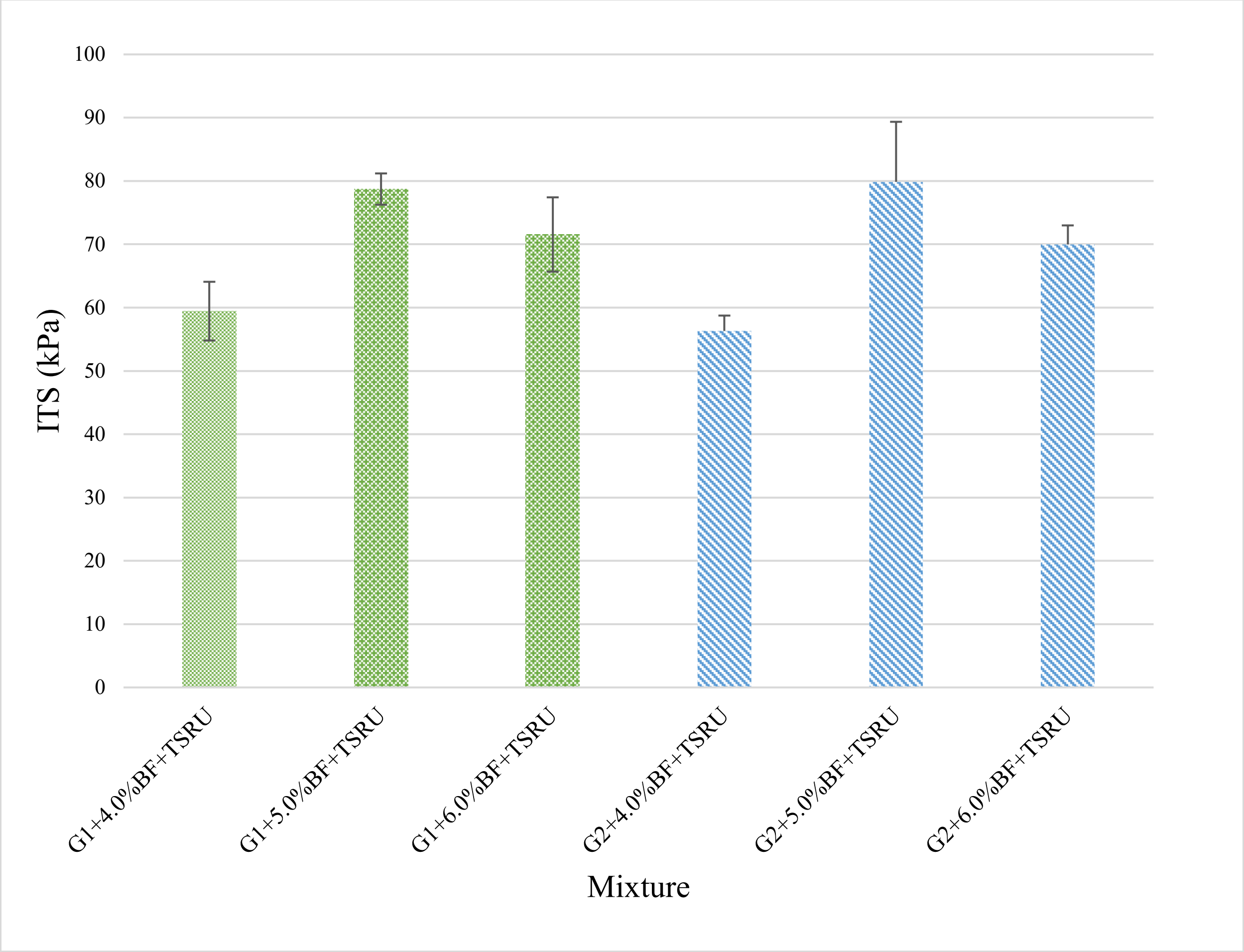

Figure 4-7 Comparison of ITS for samples with and without TSRU for both gradations.

According to the TG2 guideline [71], the minimum unsoaked ITS value for a given mixture should be 175 kPa in order for it to be considered a suitable paving mixture. For the samples prepared with aggregate Gradation 1, the highest ITS observed was 346 kPa, which satisfies the guideline. For the samples prepared with aggregate Gradation 2, the highest ITS value observed was 343 kPa, which also satisfies the guideline. According to the TG2 guideline [71], moreover, the minimum acceptable soaked ITS value is 100 kPa. The maximum soaked ITS values observed were 79 kPa and 80 kPa for Gradations 1 and 2, respectively, meaning that the minimum soaked ITS requirement was not satisfied. While we can thus conclude that the moisture sensitivity of the designed mixtures is high, this deficiency can be addressed using a suitable additive such as Portland cement [72].

4.6.2 Impact of TSRU on Tensile Strength

The ITS test results for unmodified and TSRU-modified samples with 5.0% BF were compared as illustrated in Figure 4-8. It can be observed from the results that, for both granular materials, the samples without TSRU had significantly lower ITS values in comparison with the TSRU-modified samples. The unsoaked ITS increased by 3.81 times and 2.87 times after TSRU modification for the mixtures prepared with Gradations 1 and 2, respectively. Meanwhile, the soaked ITS increased by 2.48 times and 11.96 times after TSRU modification for Gradations 1 and 2, respectively. Thus, it can be concluded from the results that the addition of TSRU improves significantly the tensile strength of mixtures.

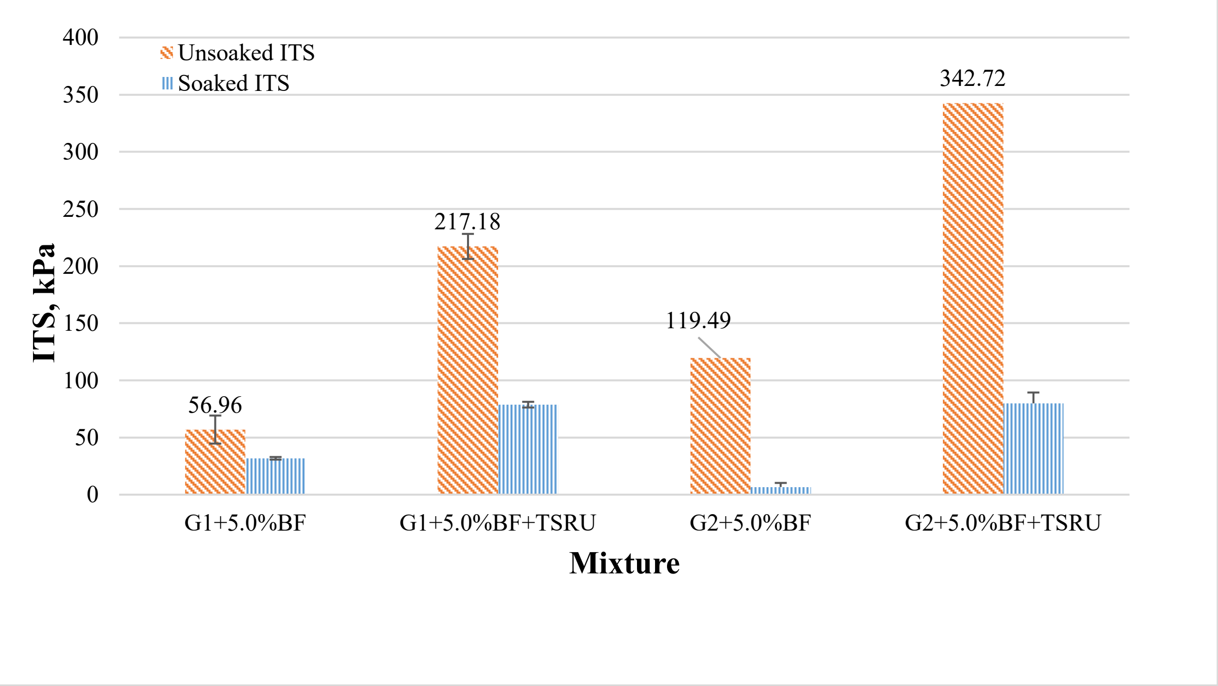

Figure 4-8 Soaked ITS test results for the TSRU-modified samples prepared with Gradations 1 and 2.

4.6.3 Estimation of Change of Thickness for Granular Layer

As per the AASTHO guide for design of pavement structures 1993 [73], the layer thickness of a pavement can be determined using equation 3-5.

$$\mathrm{D} = \frac{\mathrm{SN}}{\mathrm{a}} \qquad [4\text{-}5]$$

where

D = Thickness of granular layer (mm)

SN = Structural number

a = Layer coefficient

In reference to equation 4-5, the layer coefficient ratio before and after TSRU modification indicates the granular layer thickness ratio before and after TSRU modification. With regard to the present study, then, the layer coefficients of the untreated granular layer could be calculated from the CBR values of the aggregates, while the layer coefficients of the modified bituminous granular layer could be determined using the Marshall Stability value of the TSRU-modified samples. Thus, Marshall Stability test was conducted on the TSRU-modified samples with optimum BF content. The Marshall Stability and Flow values are presented in Table 4-4.

Table 4-4 Marshall Stability results at optimum BF content.

Gradations	**Specimen height (mm)**	**Specific gravity (G_{mb})**	**Stability (kN)**		**Flow (mm)**
			Experimental	**Corrected**	
Gradation 1	64.72	2.13	2.65	2.59	3.73
Gradation 2	64.17	2.15	3.01	3.13	3.65

Based on the CBR values of aggregate sample 1 (43%) and sample 2 (52.5%), the layer coefficients were determined to be 0.12 and 0.125 for Gradations 1 and 2, respectively. Additionally, using the Marshall Stability values, the layer coefficients were found to be 0.17 and 0.20 for Gradations 1 and 2, respectively, after modification. The ratios of granular layer thickness were 1:1.42 and 1:1.60 for Gradations 1 and 2, respectively, before and after TSRU modification. Based on these results, it can be concluded that, after modification, the thickness of the granular layer can be reduced by 42% and 60% for mixtures prepared with Gradations 1 and 2, respectively.

4.7 Conclusions

TSRU tailings and BF are byproducts of bitumen production from oil sands. This study aimed to investigate the prospect of using a water material, TSRU tailings, in base course stabilization. Comprehensive material testing and characterization were performed as the initial step, and then two types of granular materials, along with wet TSRU material and BF, were selected for the mixture design phase. Based on the results obtained, the following conclusions can be drawn:

- Comparing the two types of granular materials, sample 1 (limestone) was observed to be non-plastic, while sample 2 (alluvial) was found to be of medium plasticity and to have some clay content, meaning that sample 2 had a higher proportion of fine particles compared to Sample 1.
- The CBR test results (sample 1=43%, sample 2=52.5%) indicate that they are high quality sub-base materials and there is no significant difference in strength between the two aggregate types. Thus, both types of granular materials were used in the mix design process.
- The results of the SARA analysis indicate that the wet TSRU material has higher organic content in comparison to the dry TSRU material which indicates wet TSRU is better than

dry TSRU for improving performance properties of asphalt mixtures. Hence, wet TSRU material was used for the mix design.

- BF was found to be a relatively soft binder with a high PG of 52. Its relatively high asphaltenes content (22.67%) indicates its suitability for use as a binder in the mix design.
- Based on the ITS test results, the optimum bitumen content for the mix design was found to be 5.0%.
- The TSRU modified samples fulfilled minimum unsoaked ITS requirement, they failed to fulfil the minimum requirement for soaked ITS. Hence, the low soaked ITS value indicates moisture sensitivity of TSRU-modified samples.
- TSRU modification can increase the tensile strength of asphalt stabilized mixtures significantly. The addition of wet TSRU material was found to increase the tensile strength of the mixtures by up to 3.81 and 2.87 times for the mixtures prepared with Gradations 1 and 2, respectively.
- TSRU modification helps to decrease the granular layer thickness which indicates the cost-effectiveness of using this material in pavement design. The granular layer thickness can be reduced by up to 42% and 60% for mixtures prepared with Gradations 1 and 2, respectively, as a result of TSRU and BF modification.

Chapter 5 Investigation of Moisture Sensitivity of Granular Base Course Materials Comprised of Tailing Solvent Recovery Unit and Bitumen Froth Derived from Alberta Oil Sands.

5.1 Abstract

Tailing Solvent Recovery Unit or TSRU tailings and bitumen froth or BF are by-products derived from the processing of bitumen at Alberta oil-sands. In this research, one TSRU tailings sample, one BF sample were used to modify two granular base course materials. TSRU-modified mixtures were prepared at different concentrations of BF and their properties were investigated by indirect tensile strength or ITS test and tensile strength ratio or TSR. The main purpose of the study is to investigate the moisture sensitivity of the unmodified and modified mixtures. The obtained results showed that the TSRU modification improved the tensile strength of the granular base course materials; however, the moisture susceptibility of the modified samples was found to be high. In order to improve the moisture sensitivity of the modified samples, Portland cement was used as an additive for the modified samples and the impact of Portland cement to improve moisture susceptibility of the modified mixtures was investigated. The results indicated that the resistance of the TSRU-modified samples to moisture damage was improved after cement treatment. The cracking resistance of the modified mixtures was also evaluated using IDEAL-CT test and the results indicated that cement treatment resulted in more brittle fracture compared to nontreated TSRU modified mixtures.

5.2 Introduction

Stabilization of base course has gained popularity for improving unbound layer properties. TSRU tailings are waste by-product materials generated from oil sands bitumen production. For separating bitumen from water and solids, the paraffinic solvent is added to the froth in the froth treatment plant. The tailings containing water and solids are sent to the TSRU from the froth treatment plant to recover the paraffinic solvent. When the tailings are processed by the TSRU, they are called TSRU tailings. The TSRU tailings stream mainly contains water, asphaltenes, fines, solids, bitumen and residual solvent. Solvent content in the tailings can vary between 5 and 10%, although the exact composition of the tailings stream varies according to operating conditions [16]. TSRU tailings have higher pyrite and residual asphaltene content compared to the conventional naptha-treated froth treatment tailings [17]. These tailings can be a cost-effective alternative for stabilization of base course in pavement construction.

Bitumen Froth (BF) is also a by-product of oil sand industry generated during the production of bitumen. Froth treatment is an important step of the bitumen recovery from oil sands by surface mining. Typically, the recovered BF consists of 60 wt% bitumen, 30 wt% water, and 10 wt% mineral solid. During froth treatment process, water droplets and mineral solids are separated from the organic bitumen product by using hydrocarbon solvents but the micrometer-sized mineral particles and water-in-oil emulsion droplets are difficult to remove from the BF. The fine mineral particles are the main detriments in stabilizing the water-in-oil emulsions and the emulsified water droplets are easy to destabilize and remove in the absence of fine mineral particles [19, 21]. The treated BF can still contain on average 2−5 wt% water and 0.5−1 wt% solids [22,23].

Portland cement is generally used as an additive to asphalt emulsion stabilized mixtures. It can improve the performance properties including stiffness, strength, resistance to moisture-induced damage, rutting resistance, asphalt aggregate adhesion, and asphalt dispersion in the mixture and increase curing rate of asphalt emulsion stabilized mixtures [11, 12, 74]. A study found that addition of 1.3% cement to asphalt emulsion mixture is sufficient for significant improvement in stiffness and the ultimate resilient modulus of the modified asphalt emulsion was increased by almost 200% after adding 1% cement [75, 76]. Another study showed that the optimum cement content is between 1% and 2% by the weight of aggregates to achieve adequate performance improvement [55]. Similarly, in another investigation it was found that 1% cement by the weight of aggregates can achieve adequate resistance against cracking of asphalt stabilized mixtures in terms of increased tensile limits to failure [77].

The main objective of the research was to improve the performance of base course with the addition of TSRU tailings and BF. The tensile strength and moisture resistance property are important parameters to evaluate the performance. There were no previous studies to investigate the tensile strength and moisture resistance properties of TSRU modified mixtures for base course stabilization. Hence, to bridge this research gap, TSRU tailing, and BF were used to modify two granular base course materials. TSRU-modified mixtures were prepared at different concentrations of BF and tensile strength and moisture sensitivity of all the mixtures were investigated. In addition, the impact of Portland cement to improve the moisture resistance of modified mixtures was investigated in this research.

5.3 Objectives and Scope

The main objective of this study is to investigate the moisture sensitivity of TSRU-modified mixtures and the effects of Portland cement on improving the moisture sensitivity of these mixtures. To achieve this aim, TSRU-modified mixtures were prepared at different concentrations of BF. The tensile strength and moisture sensitivity of all the mixtures were investigated by conducting ITS and TSR tests. To improve the moisture resistance of the mixtures, Portland cement was used as an additive in the mixtures and modified samples were prepared with different cement concentration. The TSRs were compared before and after cement treatment. Additionally, in order to understand the cracking resistance of the cement treated samples, the IDEAL-CT was conducted and CT-index of the samples were compared before and after cement addition.

5.4 Materials

5.4.1 Aggregates

Two types of aggregate materials were used in this study. Aggregate sample 1 was limestone and sample 2 was gravel alluvial. These aggregates are commonly used in the construction of mine haul roads [59]. Both aggregate samples were provided by an oil sand operator. The following sections describe the properties of the used aggregate samples.

5.4.1.1 Atterberg Limit of Aggregates

The Atterberg limit test was conducted following ASTM D4318-17 [50]. Sample 1 was non-plastic because the particles did not mix with water as. For sample 2, the LL, PL, and PI were found 33%, 23%, and 10%, respectively. Normally, soils with PI within 7-17 are considered to be medium plastic and for silty clay of medium plasticity, the LL is 27-55 % and the PL is 12-33% [60]. Thus, alluvial (sample 2) has medium plasticity

5.4.1.2 Modified Proctor Test

The modified proctor test was conducted for both aggregate samples following ASTM D1557-10 [61] to determine the MDD and OMC. In case of sample 1, the MDD and OMC were found as 22.0 kN/m^3 and 5.8% respectively which were close to the values obtained for sample 2 (MDD= 22.2 kN/m^3 and OMC= 6%).

5.4.1.3 California Bearing Ratio Test

In order to measure the strength of the aggregates in wet conditions, soaked CBR test was conducted on both aggregate samples according to ASTM D1883-21 [62]. The CBR test gives an idea of the strength of a material using a standardized penetration test. The soaked CBR test results showed that sample 1 had a CBR value of 43.0% which was lower than that of sample 2 (52.5%). The CBR values indicate there is no significant difference between the aggregates in terms of strength. Therefore, both types of granular materials were used in the mix design process.

5.4.1.4 Grain Size Distribution of Aggregates

The gradations of the granular materials were determined by wet sieving following ASTM D1140-17 [63] and are provided in Figure. 5-1. Figure 5-1 shows that there is some difference between the gradations of aggregate materials but the limestone (sample 1) is finer than gravel alluvial (sample 2) and alluvial (sample 2) contains a high percentage of filler (particles smaller than 75 μm) in comparison to limestone (sample 1).

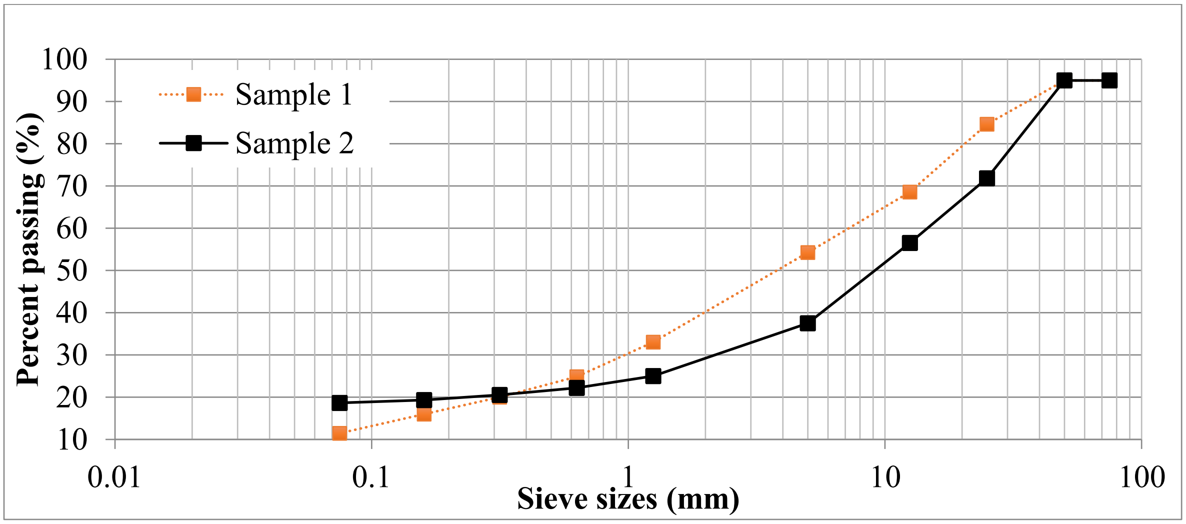

Figure 5-1 Grain size distribution of aggregate samples

5.4.2 TSRU Tailings

One type of TSRU tailing sample was used in this study. The sample remains in wet condition. It was provided by the an oil and gas company operating in the Alberta oil sands. Figure 5-2 (a) shows the TSRU tailing sample used in this research.

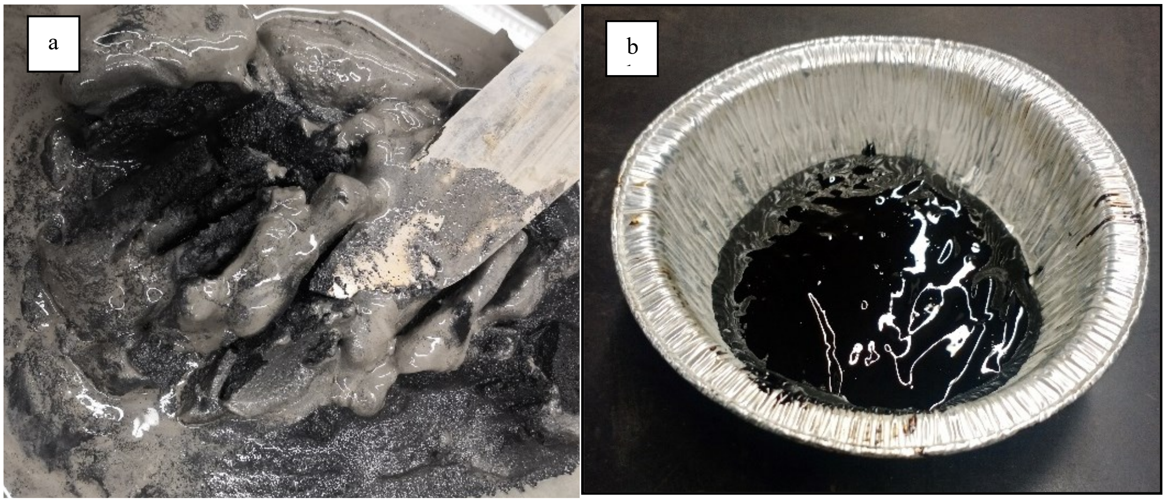

Figure 5-2 (a) TSRU tailings and (b) BF sample

5.4.2.1 Specific Gravity

The specific gravity test was conducted as per the specification of ASTM D854-17 [45] after oven-drying the sample at 110°C for 24 hours. The specific gravities of TSRU sample was found 1.78. In order to understand the changes in physical properties of TSRU materials at higher temperatures, the TSRU samples were heated at 500°C for 24 hours in an ignition oven until it reaches to a constant mass according to ASTM D7348-21 [44]. Then the specific gravity was calculated again. The results showed after ignition, the specific gravity of TSRU sample increased to 2.76.

5.4.2.2 Grain Size Distribution of TSRU Tailings

The grain size distribution of TSRU sample was determined through sieve analysis and hydrometer analysis. The gradation of the coarse particles (retained on #200 sieve) were analysed through sieve analysis and the gradation of the fine particles (passing through #200 sieve) were analysed with hydrometer analysis. The gradation was determined keeping the samples at 500°C for 24 hours (ignition method). The cumulative grain size distribution of TSRU tailings consisting of both sieve and hydrometer analysis is shown in Figure 5-3.

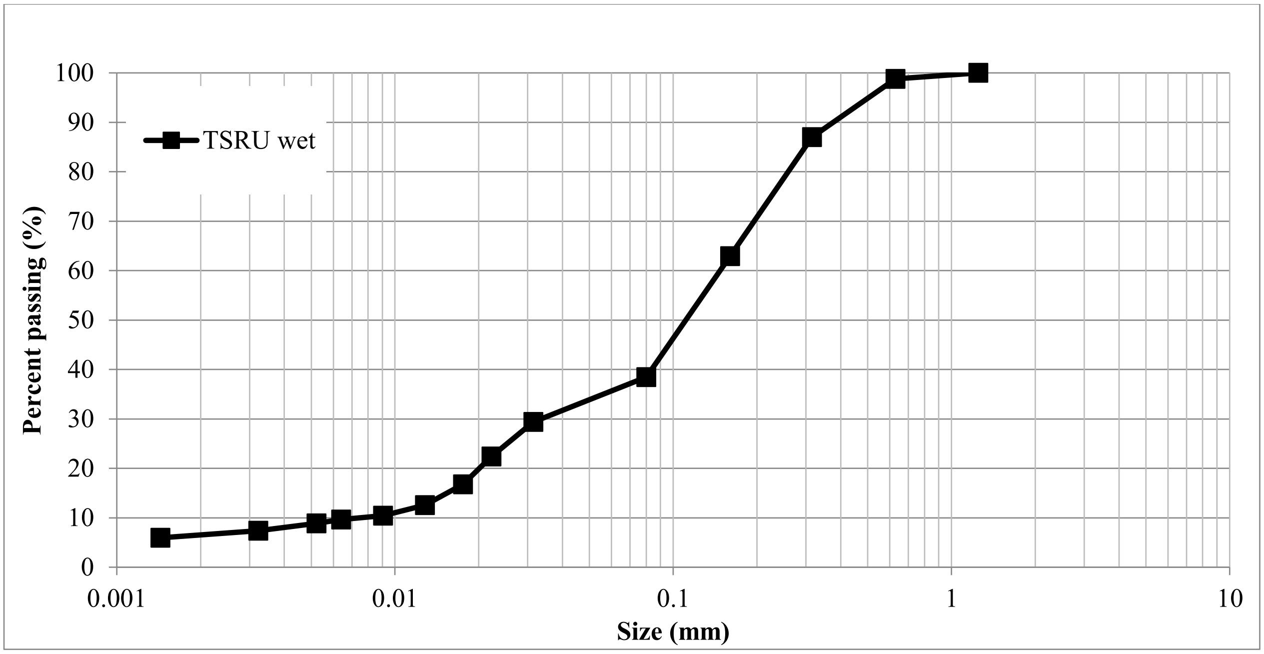

Figure 5-3 Grain size distribution of TSRU tailings sample

5.4.2.3 SARA Analysis of TSRU Tailings

The saturate, aromatic, resin and asphaltenes or SARA test was conducted using the clay-gel adsorption chromatography method following ASTM D2007-20 [48]. TSRU sample comprised of 40% organic and 60% solid after removing the percentage of water. SARA analysis showed that the TSRU sample has 10.58% saturates, 44.07% asphaltenes, 21.9% resins, and 13.66% aromatics. It was observed that the sum of the organic contents (total saturates, asphaltenes, resins, aromatics contents) was not equal to 100%. This could be attributed to the presence of inorganic salts in the sample. The solubility of the inorganic salts is very low in the organic solvents that was used in the test (Pentane, Acetone, Cyclohexane). Though the amount of the inorganic salts was not significant, the complete salt removal process is time-consuming and costly. Considering these facts, an error of 5-10% was regarded as an acceptable value.

5.4.2.4 Hydrocarbon Phase

The hydrocarbon content of a material is really important for understanding the binding property of the material. The percentage of hydrocarbon phase (asphaltenes, molten etc) of TSRU materials was calculated following ASTM D6307-19 [49] using equation 5-1. The standard is originally used to calculate the asphalt content, but here it was used to calculate the total hydrocarbon phase.

$$\% \text{ Hydrocarbon} = \left(\frac{M_A - M_B}{M_A} \times 100\right) - C_F \qquad [5\text{-}1]$$

where,

M_A = total mass of sample prior to ignition, gr

M_B = total mass of sample after ignition, gr

C_F = calibration factor ($C_F = 0$ in this case)

The hydrocarbon percentage of TSRU sample was found to be 39.69% which indicates TSRU wet is good for mixture preparation [17].

5.4.2.5 Atterberg Limits of TSRU Tailings

Atterberg limit test was conducted to understand the moisture content at which soil transition between different phases. The test result helps to get an idea about the soil type and the settlement and consolidation of the material as well. The test is done for TSRU sample following ASTM D4318-17 [50]. The values of 42%, 39%, and 3% respectively were determined for LL, PL, and PI. Generally, soils with high PI tend to be clay and soils with a lower PI value tend to have little clay. If PI is 0, the soil is non-plastic. If PI<7, the soil is slightly plastic, and if it is within 7-17, it is medium plastic, and if PI>17 then it is highly plastic [53]. The PI of TSRU wet was found to be

3% (<7) which indicates TSRU wet is slightly plastic and it is mainly silt and has very low clay content. However, a high PI generally indicates a low shear strength and the higher the plasticity, the more shrinkage occurs during drying. On the other hand, a low PI indicates that the soil will change significantly in consistency even with a small change in water content [52]. Therefore, as TSRU wet has low plasticity, its shrinkage is not expected to be very high although it could be very sensitive to moisture.

5.4.2.6 Methylene Blue Index (MBI) Test

The Methylene Blue Index (MBI) test provides an indication of the clay activity of soil. The MBI test was conducted on TSRU wet sample following ASTM C837-19 [53] to estimate the clay content present in it. The average MBI value of the TSRU wet sample was calculated using equation 5-2. The average value of three replicates showed that the MBI of TSRU wet is 1.73. For oil sand clay, the expected value of MBI for the pure sample is 14 [54]. Comparing with it, MBI of the TSRU wet was found low. For quantifying the clay content in TSRU wet, the % clay in TSRU wet was calculated using equation 5-3. Clay amount in TSRU wet was found to be 12.6% which indicates TSRU wet can be sensitive to moisture.

$$\text{MBI (meq/100g)} = (\text{mls in MB} \times \text{Normality of MB})/(\text{gr of samples}) \times 100 \qquad [5\text{-}2]$$

$$\%\ \text{clay} = [\text{MBI Meq/100gr} + 0.04\ ]/\ 0.14 \qquad [5\text{-}3]$$

5.4.3 Bitumen Froth

BF is a by-product produced in the Alberta oil sand industry. The binder is very soft, and it contains a small amount of water-oil emulsion. In this study, one type of BF sample was provided by an oil sand operator and used along with granular materials and TSRU wet for the mixture preparation. Figure. 5-2 (b) shows the BF sample that was used for this research.

5.4.3.1 SARA Analysis of Bitumen Froth

The SARA test was conducted on BF to understand the amount of four important hydrocarbon groups (saturates, asphaltenes, resins, and aromatics) which helps to predict the polar and non-polar nature of the material. The test revealed that BF is composed of 16.54% saturates, 22.67% asphaltenes, 32.84% resins, and 23.76% aromatics. The asphaltene content indicates BF can be used as a binder for the mix design [17].

5.4.3.2 Viscosity

The viscosity of asphalt binders gives idea about the fluidity and workability of a binder. It also can be used to determine the mixing and compaction temperatures of asphalt mixtures. In accordance with AASHTO T 316-19 [55], a Brookfield rotational viscometer was used to measure the viscosity of the BF binder at different temperatures. Table 5-1 shows the viscosity of the BF at tested temperatures.

Table 5-1 Rotational viscosity of BF at different temperatures

Temperature (°C)	Viscosity (Pa.s)
70	3.48
80	1.72
90	0.95
100	0.57

According to the Federal Highway Administration (FHWA) [56], compacting asphalt mixtures requires mixing and compaction under equiviscous temperature conditions corresponding to 0.170 ± 0.020 Pa.s for mixing and 0.280 ± 0.030 Pa.s for compaction. It is worth mentioning that as the

BF contains water, the test temperature could not be raised to more than the boiling point of water (100°C) to ensure the consistent material property. By extrapolating the values, the mixing temperatures were found to be more than 100°C but due to the limitation of raising the temperature, 95°C was selected as mixing temperature based on engineering judgment.

5.4.3.2 Rheology Test

To understand the high-temperature properties of the BF, a rheology test was conducted on unaged and aged BF samples. A Dynamic Shear Rheometer (DSR) was used for the rheology test according to AASHTO T 315-20 [57]. In this test, a spindle with the diameter of 25 mm and 1 mm gap was used for the unaged samples. To simulate asphalt binder aging in the plant, the asphalt samples were aged using a Rolling Thin-Film Oven (RTFO) based on AASHTO T240-21 [58]. According to AASHTO M320-21 [70], when testing 25 mm-diameter samples at high temperatures, the temperature at which the parameter $G^*/\sin\delta$ drops below 1.0 kPa or 2.2 kPa, was recorded as the failure temperature for unaged and RTFO-aged binder, respectively. The minimum temperature among those two was the final continuous high-performance grade (PG) of the binder. The high-PG for unaged binder was found to be 52°C whereas for RTFO-aged binder was 56.7°C. Thus, the continuous PG grade test result was 52°C. The high-PG grade indicates the BF can meet the high-PG grade criteria for colder climatic regions like Alberta, Canada.

5.4.4 Portland Cement

Portland cement meeting the specifications of ASTM C1157 [31] was used in this study. It was used as an additive in the mix design. The cement samples were obtained in powder form. In terms of its basic properties, the cement had an initial setting time of 45 min, a final setting time of 420 min, and a compressive strength of 13 MPa at 3 days, 20 MPa at 7 days, and 28 MPa at 28 days.

5.5 Methodology

5.5.1 Preparation of Modified Mixture

In this study, aggregate samples 1 and 2 were used for separate mix designs. These gradations were kept consistent for all subsequent mixtures. The OMC values of both aggregate types were determined from the modified proctor test. The OMC values were found to be 5.8% and 6.0% for samples 1 and 2 respectively. At first, both aggregate samples were oven-dried at 110°C for 24 hrs and crushed using a wooden hammer. Only the particles passing through a 20-mm sieve were selected for the preparation of the mixtures. The water requirement for the mixture was calculated based on the OMC of the aggregates found from the modified proctor test. During the mixing process, no external water was added, rather the water of TSRU sample was used to reach the OMC. Based on the optimum water requirement, the required TSRU content was calculated. The mixing process was initiated by mixing aggregates (25°C) with TSRU sample (95°C). Thereafter, hot BF (95°C) was added to the mixture. The BF was heated at 95°C because of the presence of water in it. The samples were mixed until the TSRU materials and BF were uniformly distributed throughout the aggregate blend. The material proportions for the mix design are provided in Table 5-2 and the modified gradations for aggregate samples 1 and 2 are shown in Figure 5-4. The samples were prepared using 75 blows of Marshall hammer per each side of the sample. For ITS test on unsoaked specimens, three replicates were prepared for BF contents of 0 to 6% by 1% intervals and for ITS test on soaked specimen, three replicates for 4%, 5% and 6% contents were also made. Finally, the samples were put into an oven for curing at 40°C for 72 hrs as specified by the TG2 guideline [71]. The cured samples were then kept at room temperature for at least 2 hr prior to extraction from the mold.

Table 5-2 Materials proportion for mix design

Materials	Gradation 1	Gradation 2	Comment
Total aggregates (g)	1,100	1,100	
Bitumen froth (g)	44 (4% of total aggregates)	44 (4% of total aggregates)	Depends on percentage of BF
Required water (g)	63.8	66	Based on optimum moisture content of aggregates
Moisture drop in TSRU (%)	20	20	Heated at 95°C for 2h
Required TSRU (g)	224	217	Based on the required water
Asphaltenes in mix (%)	1.6	1.7	Only the powder asphaltenes in TSRU (44.07%) was considered here.

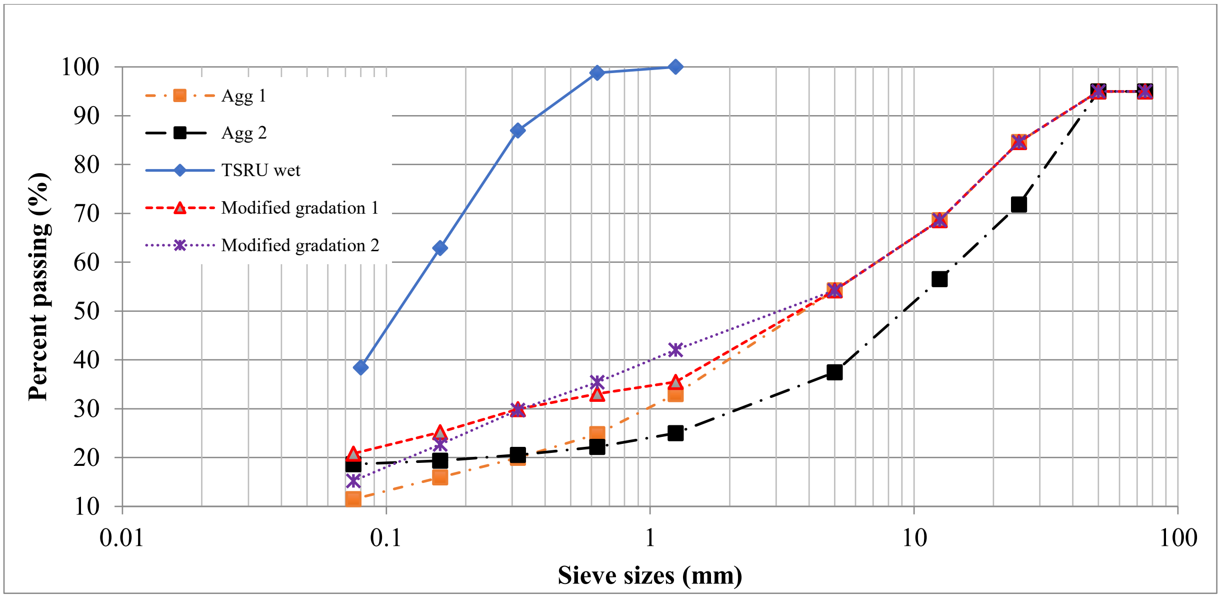

Figure 5-4 Modified gradation for aggregate samples 1 and 2

5.5.2 Indirect Tensile Strength Test

The ITS was performed according to AASHTO T 283-21 [38] on the designed samples. For the unsoaked subset, the ITS test was conducted on dry samples containing BF content of 0 to 6% by 1% intervals. The ITS test was also conducted on the saturated (soaked) samples containing 4%, 5% and 6% BF contents in order to determine the optimum BF. For the saturated samples, the samples were conditioned in water at 25°C for 24 hrs. The ITS tests were conducted by applying compressive load at a rate of 50 mm/min. During the test, the maximum load applied to the sample until failure was recorded in order to calculate the tensile strength of the samples. The strength of each sample was calculated as per equation 5-4.

$$S_t = (2000*P)/(\pi*t*D) \qquad [5\text{-}4]$$

where

S_t = indirect tensile strength, kPa

P = maximum applied load, N

t = average height of specimen, mm

D = diameter of specimen, mm

The ITS values were calculated in different BF contents and from the result, the optimum BF content (where maximum ITS value is found) for the mix design.

5.5.3 Moisture Susceptibility

The ITS test was conducted on the saturated samples containing 4%, 5% and 6% BF contents. The samples were conditioned in water at 25°C for 24 hrs before conducting the ITS test on soaked samples. The TSR for the saturated samples were determined using equation 5-5.

$$TSR = S_2/S_1 \qquad [5\text{-}5]$$

where

S_1 = Average tensile strength of the dry subset (kPa)

S_2 = Average tensile strength of the saturated subset (kPa)

The TSRs of the modified samples indicates the moisture sensitivity of the TSRU modified samples. Low TSR indicates high moisture sensitivity. If the moisture sensitivity of modified samples is high, this property needs to be improved.

5.5.4 Preparation of Mixtures Modified with Portland Cement

In order to investigate the impact of Portland cement to improve the moisture resistance of the modified samples, Portland cement was added in the mix design and samples were prepared for 0.5%, 1%, 2% and 3% cement by total weight of the mixture with optimum BF content . The cement was mixed with oven-dried aggregates after the aggregates were cooled to room temperature. Then TSRU and BF were heated at 95°C and added to the mixture and mixed until a uniform mixture was achieved similar to the previous mix design. The curing process was kept similar as explained before. ITS test was conducted on both unsoaked and soaked samples.

5.5.5 IDEAL-CT Analysis

The IDEAL-CT test was conducted for both cement-modified and unmodified mixtures as per ASTM D8225-19 [41]. To calculate the CT- Index, the ITS test was performed on 0%, 0.25%, 0.5%, 1%, 2% and 3% dry samples at the same loading rate as described above. Once the load versus displacement curve of each specimen from ITS test had been obtained, the CT Index was calculated using equation 5-6.

$$\mathbf{CT_{Index}} = \frac{\mathbf{t}}{\mathbf{62}} * \frac{\mathbf{G_f}}{|\mathbf{m_{75}}|} * \frac{\mathbf{l_{75}}}{\mathbf{D}} \qquad [5\text{-}6]$$

Where

G_f = fracture energy (kN/mm) which is determined from the ratio of the area under the load vs. displacement curve divided by the product of the thickness (t) and diameter (D);

l_{75} = post-peak displacement rate at 75% of the peak load (mm);

$|m_{75}|$ = slope of the post peak curve at 75% of the peak load (kN/mm);

D = specimen diameter (mm); and

t = specimen thickness (mm).

5.6 Results and Discussion

5.6.1 Indirect Tensile Strength Test

The ITS test results for the TSRU-modified unsoaked samples for both aggregate samples are presented in Figure 5-5. According to the the TG2 guidelines [71], the minimum ITS value for unsoaked samples should be 175 kPa for a mixture to be considered as a paving material. For the aggregate sample 1, the highest ITS of the TSRU-modified mixture was found 346 kPa which is satisfactory in accordance with the guideline. For aggregate sample 2, the highest ITS value was 343 kPa which also fulfils the minimum requirement.

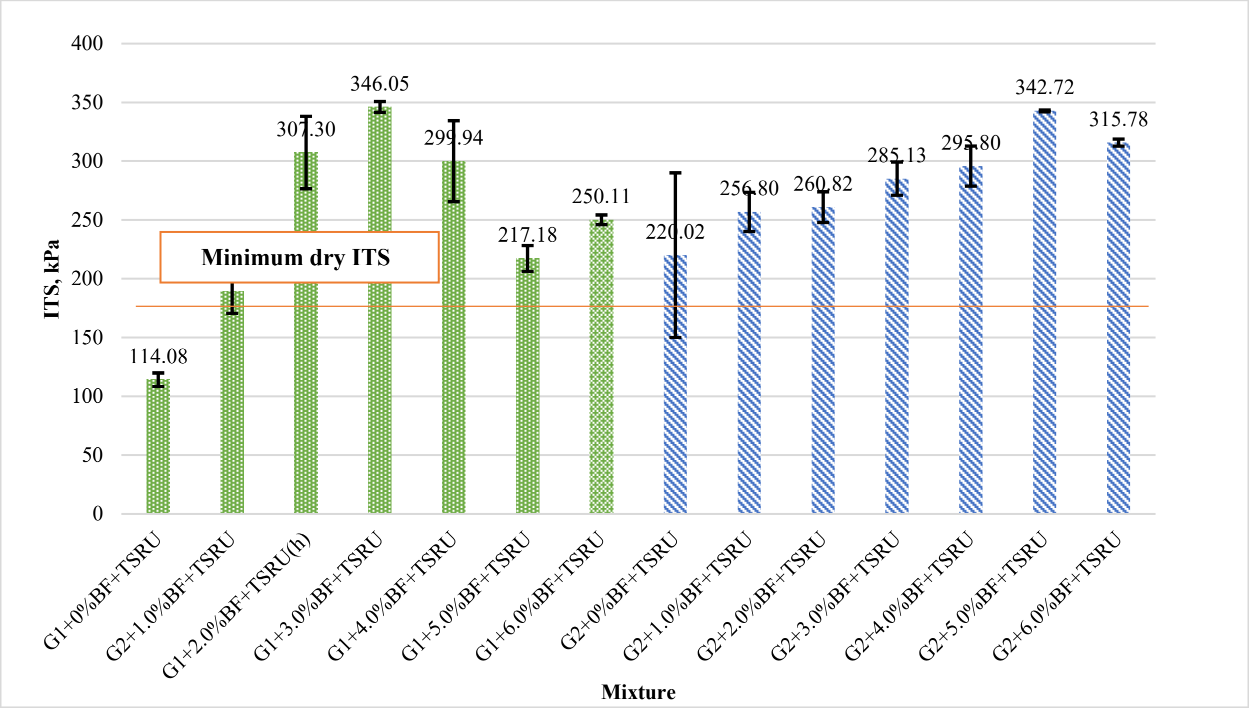

Figure 5-5 ITS test results for unsoaked TSRU-modified samples

The TSR can be used to evaluate the moisture sensitivity of asphalt mixtures. The ITS results on soaked samples and TSR values are presented in Figure 5-6. As shown in this figure, the maximum ITS value for soaked sample was found for 5% BF content for both gradations. Hence, 5% BF was regarded as the optimum BF content for this mix design.

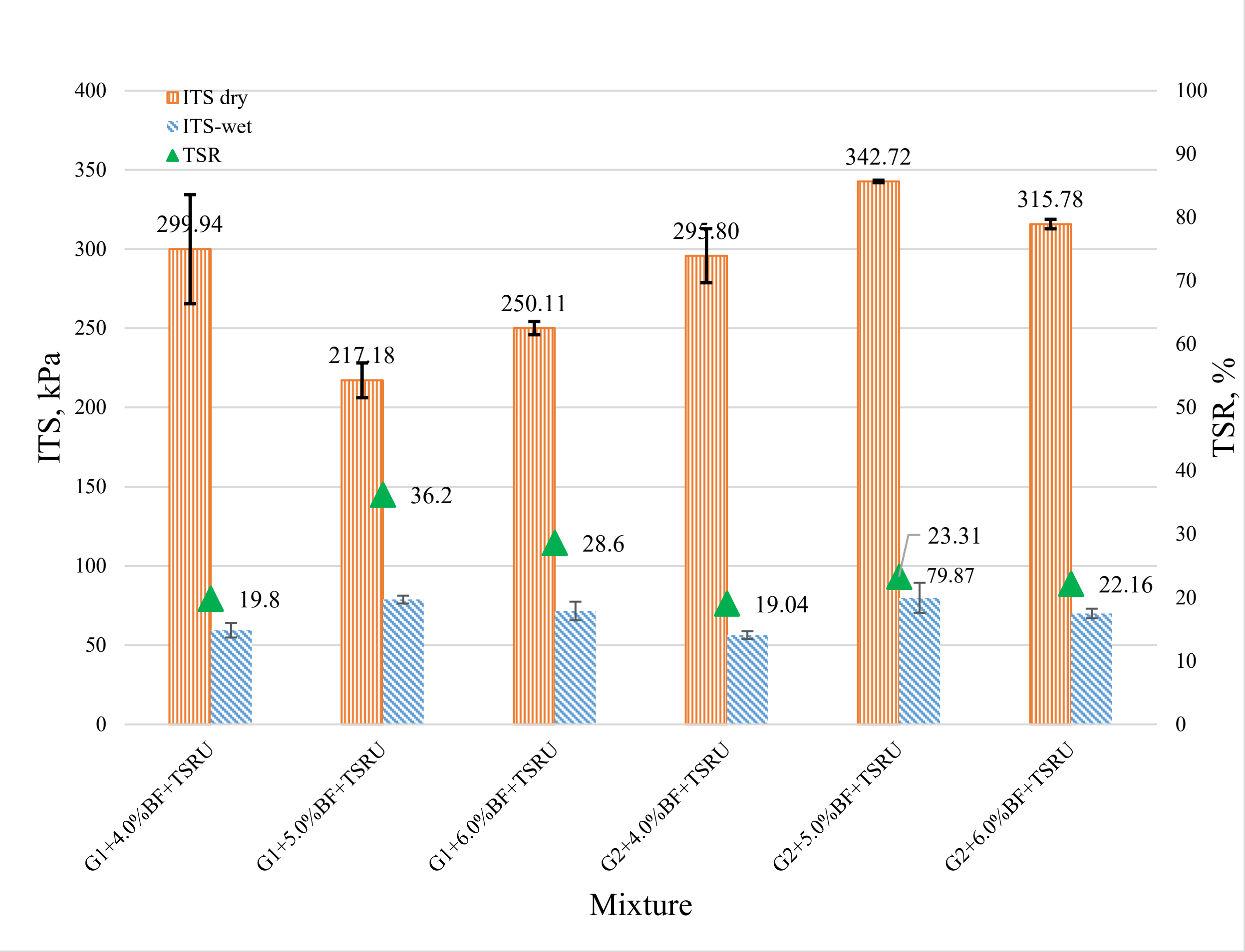

Figure 5-6 Moisture susceptibility of TSRU-modified samples from ITS test

According to the TG2 (2020) guidelines [71], the minimum acceptable soaked ITS value should be 100 kPa. The maximum ITS values were 79 kPa and 80 kPa for soaked samples fabricated with aggregates 1 and 2 respectively. It was clear that ITS requirement was achieved by the mix designs for unsoaked samples but the minimum ITS requirement was not satisfied for soaked samples. Moreover, the modified samples fabricated with aggregate 1 had higher TSR compared to samples fabricated with aggregate 2 indicating higher resistance to the moisture-induced damage. Therefore, it could be concluded that the moisture sensitivity of the designed mixtures was high. The possible reason of lower moisture resistance of gradation 2 could be due to the presence of clay in the aggregate material. As clay particles are unstable and very sensitive to variation of humidity [78], the mixtures having clay may experience shrinkage and lose some of its strengthHowever, the moisture resistance can be improved by addition of different additives such as Portland cement [72]. So, Portland cement was added as an additive in the mix design and ITS test was conducted on the cement treated samples.

5.6.2 Comparison between Cement-Modified and Unmodified Samples

In order to improve the moisture sensitivity of the TSRU-modified samples, 0.5%, 1%, 2% and 3% Portland cement-treated samples were prepared for both soaked and unsoaked conditions of the ITS test. The main goal was to improve the ITS value for soaked samples to fulfil the minimum requirement (100 kPa). The results are presented in Figure 5-7. From the results, it can be observed that for samples fabricated with aggregate 1, the addition of cement can help to fulfil the minimum soaked ITS requirement. The tensile strength can be increased by 41% after 3% cement treatment for samples fabricated with aggregate 1. For samples fabricated with aggregate 2, the minimum ITS requirement for soaked sample can be achieved after 2% and 3% cement content and the tensile strength can be increased by 26% after 3% cement modification. 0.5% cement treated samples fabricated with aggregate 1 can be considered more economic option than 2% cement treated samples fabricated with aggregate 2. Overall, it can be concluded that after 0.5% and 2% cement treatment, the modified samples fabricated with aggregates 1 and 2 can fulfil the minimum ITS requirement for soaked samples respectively.

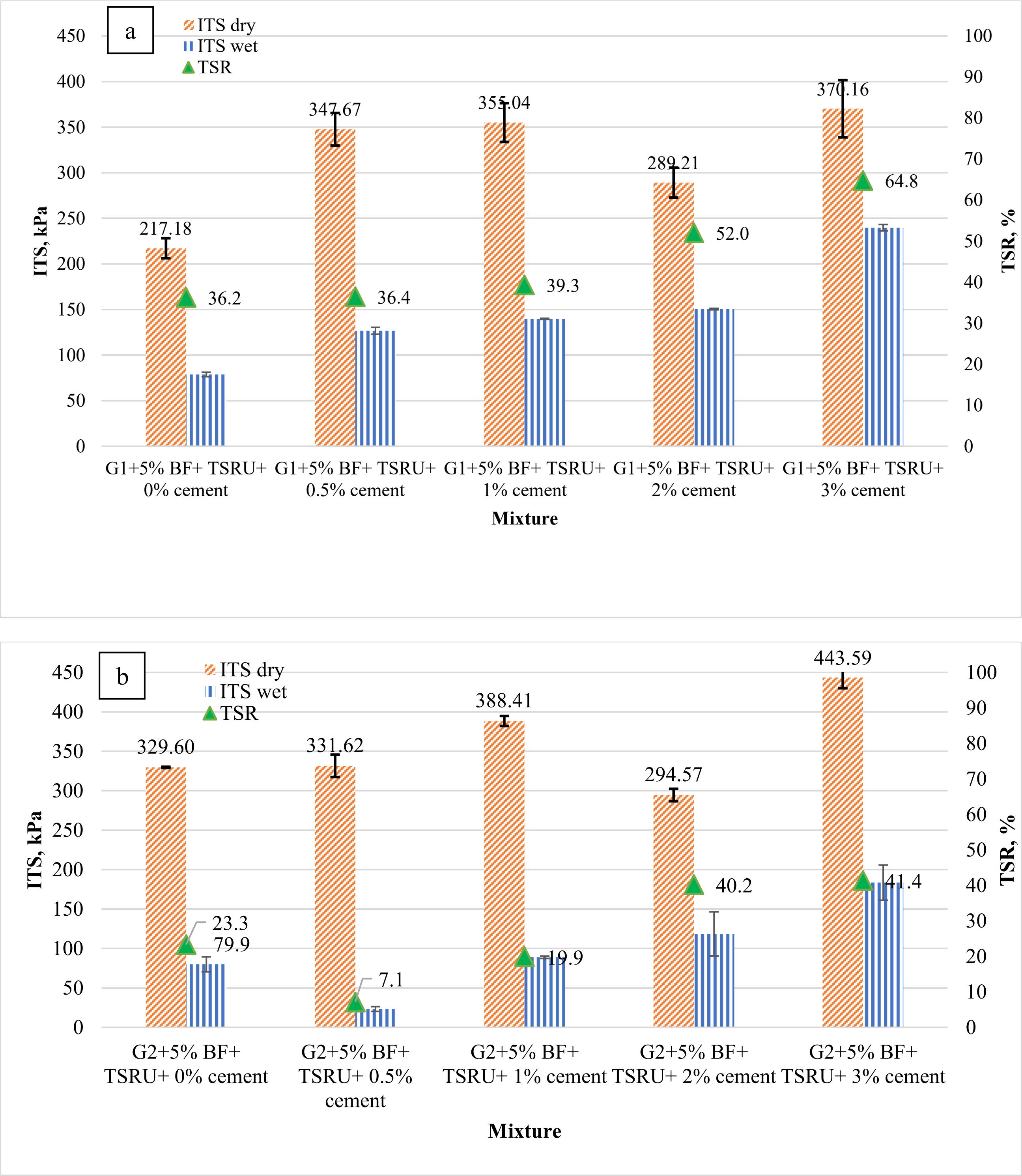

Figure 5-7 ITS result comparison of cement-modified samples fabricated with (a) aggregate 1 (b) aggregate 2

5.6.3 IDEAL-CT Analysis

The load-displacement graphs for the ITS test results are shown in Figure 5-8 which were used to estimate the CT-index of the mixtures. Figure 5-8 (a) presents the load-displacement graphs of the cement treated samples fabricated with aggregate 1, where the slope of the plot or displacement rate after the pick load is indicative of how rapidly a crack will propagate in each the mixtures once initiated. It can be seen that the slope after the peak point is steeper in the cement-treated samples than in the untreated sample, indicating more rapid crack propagation. Figure 5-8 (b) shows similar characteristics indicating the rapid crack propagation of cement treated samples fabricated with aggregate 2 as well. It can also be observed that the graph of the cement treated samples fabricated with aggregate 1 is flatter than that of the cement treated samples fabricated with aggregate 2 indicating higher failure energy for cement treated samples fabricated with aggregate 2.

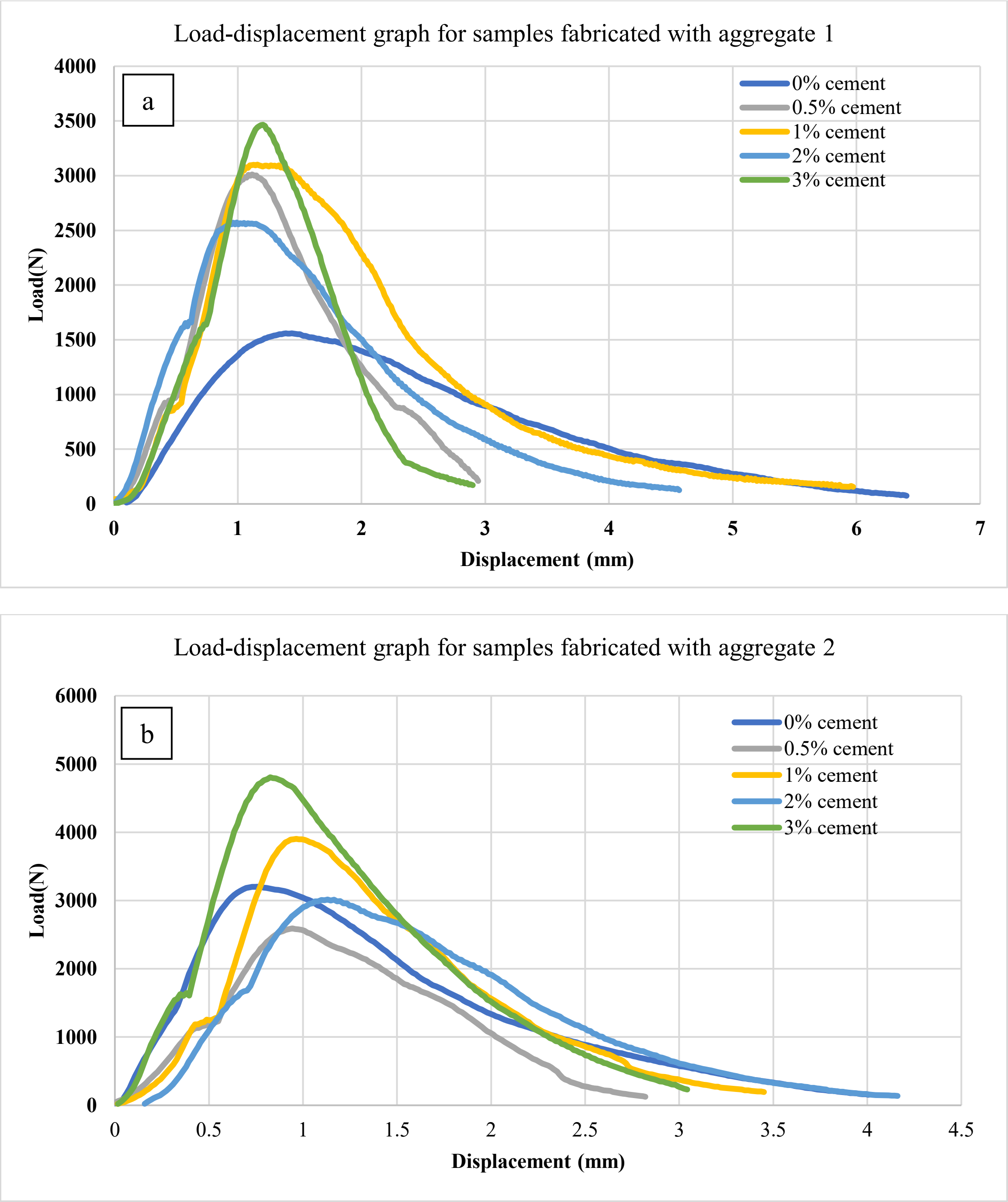

Figure 5-8 Load-displacement graphs of cement-modified samples fabricated with (a) aggregate 1 (b) aggregate 2

The CT-Index is a good performance indicator for cracking resistance [62]. Hence, in this research the CT-index and fracture energy values were calculated in accordance with ASTM D8225-19 [62]. The CT-Index and fracture energy for both aggregate types are compared in Figures. 5-9 and 5-10. From the results, it can be concluded that the addition of cement affected the CT-index values significantly. The overall trend indicates with the increase of cement content, the CT-index decreases which represents lower cracking resistance for the samples with a high cement content. The maximum CT-index reduction was obtained for 3% cement content for both aggregate types. The maximum CT-index reduction relative to the untreated samples were 85% and 64% for cement treated samples fabricated with aggregate samples 1 and 2 respectively. Comparing the FE values of the modified mixtures, the increase in FE was higher in the modified mixtures fabricated with aggregate 2 than in the modified mixtures fabricated with aggregate 1. Overall, it can be concluded that the cement treatment makes the mixture more brittle and increases the chance of brittle fracture in the mixtures.

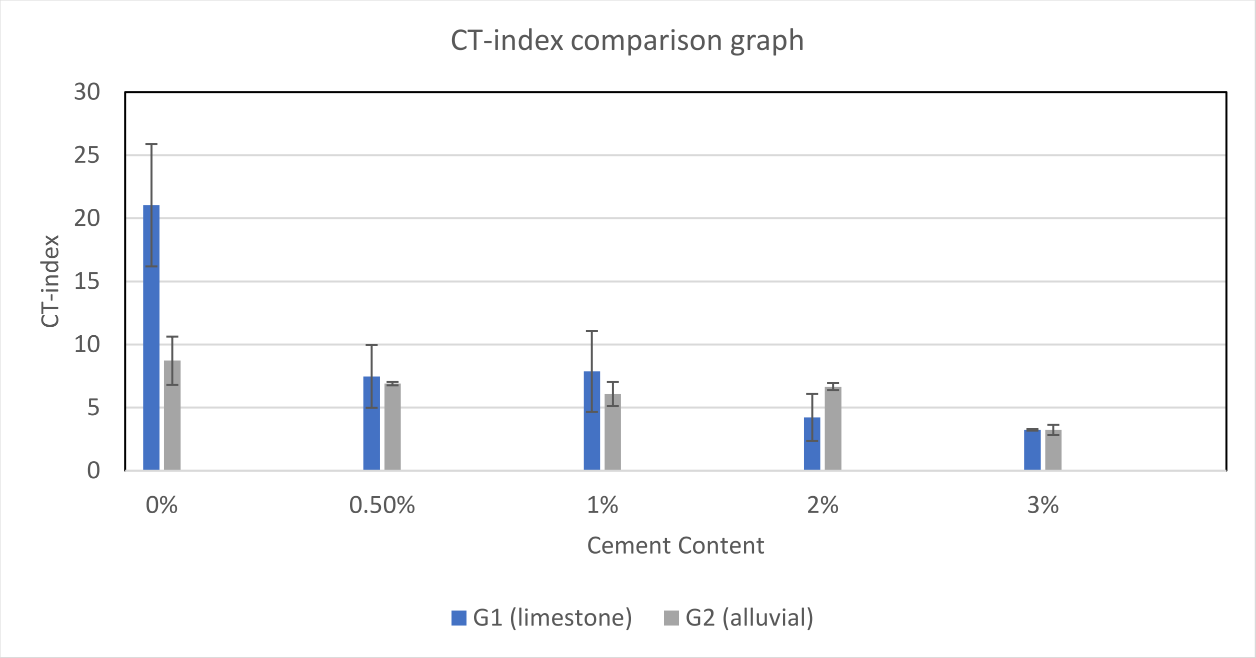

Figure 5-9 CT-index comparison of cement-modified samples

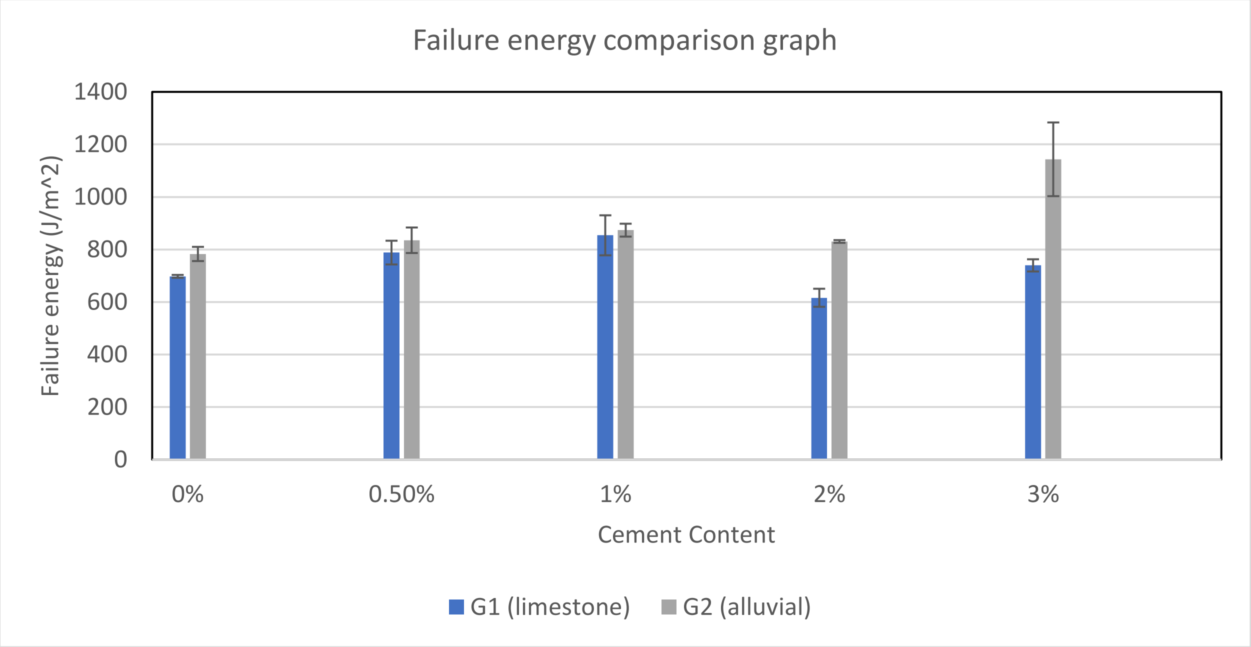

Figure 5-10 Fracture energy comparison of cement-modified samples

5.7 Conclusions

TSRU tailings and bitumen froth are by-products produced during Alberta oil sand bitumen production. This focus of this study was to investigate the moisture sensitivity of TSRU modified samples and improve this property through the Portland cement treatment. A comprehensive material testing, and characterization were performed initially, and mixtures were prepared with two types of granular materials along with TSRU tailings and BF. Based on the results obtained in this study, the following conclusions can be derived:

- Comparing the granular materials used in this study, it was observed that aggregate 1 (limestone) was non-plastic, although aggregate 2 (alluvial) had medium plasticity and some amount of clay. Consequently, aggregate 2 had higher fine particles compared to aggregate 1. On the other hand, California Bearing Ratio (CBR) test results of the granular materials indicated that there is no significant difference in strength between the two aggregate types. Therefore, both types of granular materials were used in the mix design process.
- SARA analysis indicated TSRU tailings had higher organic content present in it. Hence, it was a good option to be used in the mix design.
- BF was found as a soft binder with a high PG of 52°C which can be considered as a suitable binder for colder regions where the pavement temperature does not increase much during the summertime.
- The maximum strengths of the unsoaked TSRU-modified mixtures were found to be 346 kPa and 343 kPa for samples fabricated with aggregate samples 1 and 2 respectively which fulfils the minimum requirement of 175 kPa. On the other hand, the maximum strengths of

the soaked specimens were 79 kPa and 80 kPa for samples fabricated with aggregates 1 and 2 respectively which fails to fulfil the minimum requirement of 100 kPa.

- ITS test results of cement treated samples indicated after 0.5% and 2% cement treatment, the modified samples fabricated with aggregates 1 and 2 can fulfil the minimum soaked ITS requirement respectively.
- The tensile strength could be increased by 41% and 26% after 3% cement modification for samples fabricated with aggregates 1 and 2 respectively.
- Cement treatment had an adverse effect on the cracking resistance of TSRU modified mixtures. The maximum CT-index reduction relative to the untreated samples were 85% and 64% for cement treated samples fabricated with aggregate samples 1 and 2 respectively.
- Comparing the fracture energy of the cement treated mixtures, the increase in fracture energy was higher in the modified mixtures fabricated with aggregate 2 than in the treated mixtures fabricated with aggregate 1.

Chapter 6 Summary and Conclusions

6.1 Summary

TSRU tailings are waste by-products from Alberta oil sands which has no significant use anywhere. On the other hand, bitumen froth is also produced during oil sands bitumen production. Application of TSRU tailings and bitumen froth to stabilize base course can be a new approach in pavement construction to enhance the performance of pavement layers. There was no publicly available study before about using TSRU tailings and bitumen froth as stabilizing agents in pavement construction. This study is a pioneer one in this respect and it results in an opportunity to investigate the impacts of the waste TSRU materials in improving the performance of base course layer of pavement. Because of the cost-effectiveness, TSRU materials can be regarded as potential stabilizing agents for base course stabilization in pavement construction.

In this study, the characteristics and properties of the TSRU tailings, bitumen froth and granular material samples were investigated by various tests based on Superpave testing protocols as well as AASHTO/ASTM standards. The main focus of this research study is to investigate the impact of TSRU tailings and bitumen froth modification to improve the granular layer properties and reduce the thickness of road with no additional costs. The tensile strength and moisture sensitivity of the stabilized mixtures are thoroughly investigated. The change of thickness of granular layer is also estimated in accordance with AASHTO-1993 guideline. Portland cement are also used as an additive later into the mixtures to analyze and improve the moisture resistance property of TSRU modified mixtures. The cracking resistance of modified mixtures before and after cement treatment is also compared in the study. The result of the study will be helpful for the pavement industry for introducing a new concept of using waste materials to enhance the granular layer properties and reduce the thickness of granular layer.

6.2 Conclusions

Different tests on TSRU tailings modified asphalt stabilized mixes were conducted and the conclusions drawn from the study are summarized as follows:

- Comparing the characteristics of two types of granular materials used in this study, it was found that limestone was non-plastic, while alluvial had medium plasticity and some clay content, meaning that alluvial had a higher proportion of fine particles compared to limestone. Moreover, the CBR test results (limestone=43%, alluvial=52.5%) indicate that there is no significant difference in strength between the two aggregate types. Thus, both types of granular materials were used in the mix design process.
- The results of the SARA analysis indicate that the wet TSRU tailings has higher organic content in comparison to the dry TSRU tailings which indicates wet TSRU is better than dry TSRU for improving performance properties of asphalt mixtures. Hence, wet TSRU tailings was used for the mix design.
- BF was found to be a relatively soft binder with a high PG of 52. Its relatively high organic content including asphaltenes (22.67%) and performance grade indicates its suitability for use as a binder in the mix design in cold region.
- Based on the ITS test results of TSRU modified samples, the optimum bitumen content for the mix design was found to be 5.0%.
- Although the TSRU modified samples fulfilled minimum unsoaked ITS requirement, they failed to fulfil the minimum requirement for soaked ITS. Hence, the low soaked ITS value indicates moisture sensitivity of TSRU-modified samples.
- TSRU tailings modification can increase the tensile strength of asphalt stabilized mixtures significantly. The addition of wet TSRU tailings was found to increase the tensile strength

of the mixtures by up to 3.81 and 2.87 times for the mixtures prepared with limestone and alluvial, respectively.

- From CBR and Marshall Stability tests, it was confirmed that TSRU modification helps to reduce the granular layer thickness. The granular layer thickness can be reduced by up to 42% and 60% for mixtures prepared with limestone and alluvial, respectively, as a result of TSRU and BF modification. It indicates TSRU tailings and BF modification can be a cost-effective solution in pavement design.
- After adding cement as an additive in TSRU modified mixtures the moisture resistance of modified samples improves. The ITS results of cement modified samples indicate after 0.5% and 2% cement modification, the modified samples fabricated with limestone and alluvial can fulfil the minimum soaked ITS requirement respectively.
- Cement addition can improve the tensile strength of the modified samples as well. The tensile strength can be increased by 41% and 26% after 3% cement modification for samples fabricated with limestone and alluvial respectively.
- Cement modification can have an adverse effect on the cracking resistance of TSRU modified mixtures. The maximum CT-index reduction was found 85% and 64% for cement samples fabricated with limestone and alluvial respectively in comparison to the cement-unmodified samples was.
- Comparing the fracture energy of the cement modified mixtures, the increase in fracture energy was higher in the modified mixtures fabricated with alluvial than in the modified mixtures fabricated with limestone.

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