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Feasibility of Using Combustion-Based Methods to Quantify Saline-Based Anti-Stripping Agent in Modified Asphalt Binders

Riyadul Hashem Riyad [1], Ji Wu [2] and Junan Shen [1,*]

[1] Civil Engineering and Construction, Georgia Southern University, Statesboro, GA 30458, USA; rr14527@georgiasouthern.edu

[2] Department of Biochemistry, Chemistry and Physics, Georgia Southern University, Statesboro, GA 30458, USA; jwu@georgiasouthern.edu

* Correspondence: jshen@georgiasouthern.edu; Tel.: +1-9124780084

Abstract: "Anti-stripping Agents" or "adhesion promoters" can enhance the chemical affinity between asphalt and aggregate by increasing their mutual attraction. Various forms of anti-stripping agents have been proposed to mitigate pavement stripping, and siloxane-based Zychotherm is one of them. Choosing the appropriate type and dose of anti-stripping additives is no doubt vital to the intended performance. Therefore, it is critically important to determine the dose of the additives used in the modification of asphalt binders. This research developed a feasible detection method that can closely measure the dose (0.05% and 0.1%) of siloxane-based anti-stripping liquid agents. Related test methods, including heat combustion test, residue visualization, burning, and ignition, were implemented. The heat combustion results showed that with the addition of the Zychotherm anti-stripping additive, the average heat combustion value decreased by 1.34% and 1.72% for 0.05% and 0.1% Zychotherm-modified binder, respectively. In the burning and ignition process, the modified binder left yellowish substances in the residue, which is an indication of the presence of Zychotherm. The weight of the yellowish residue related more to the quantity of Zychotherm in the asphalt binder.

Keywords: asphalt; Zychotherm; heat combustion; siloxane-based; ignition; residue

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1. Introduction

The trend of urbanization in North America is on a continuous rise, underscoring the imperative need for robust transportation infrastructure to accommodate the burgeoning urban population. A 2018 United Nations study revealed that over 82% of the continent's populace resided in urban areas, a figure that is projected to escalate to 90% by the midpoint of the 21st century [1]. This demographic shift accentuates the critical role of maintaining and enhancing highways to support the increasing demands on transportation systems. As urban centers grow, the efficient movement of people and goods becomes increasingly vital, making the investment in and upkeep of road infrastructure a priority for ensuring economic vitality and quality of life [2].

In the realm of road construction materials, asphalt pavement stands out as a preferred choice across North America due to its myriad advantages. Known for its low noise emission, exceptional wear resistance, ample availability of raw materials, remarkable strength, reduced fuel consumption, and enhanced driving comfort, asphalt offers a flexible and effective solution for roadway [3]. This preference is evidenced by the extensive utilization of asphalt mixtures in the United States, where they account for nearly 94% of the nation's road surfaces. Similarly, in Canada and Mexico, the reliance on asphalt for road construction is notably high, with about 90% and 96% of roads, respectively, constructed using this material [4]. Such widespread adoption underscores asphalt's role as a cornerstone in the development and maintenance of North America's transportation infrastructure, facilitating smoother and more reliable travel for millions.

Most pavements in humid and wet regions experience failures such as rutting and stripping [5]. These failures are caused by factors such as traffic stress, hot temperature, and water [6–8]. The greater affinity of the aggregate surface for water, as compared to asphalt, is regarded as the primary catalyst for the stripping phenomenon in asphalt mixtures [9,10]. The adhesion between bitumen and aggregates plays a crucial role in determining the stability of flexible pavement. The problem of stripping, which varies depending on the kind of aggregates, can be attributed to the ionic nature of the aggregates [11–13]. The chemical affinity between asphalt and aggregate can be enhanced by introducing minute amounts of chemicals that alter the properties of either the asphalt or the aggregate, thus increasing their mutual attraction. These substances are commonly referred to as "Anti-stripping Agents" or "adhesion promoters" [14,15].

From a technical perspective, various forms of anti-stripping agents have been proposed to mitigate pavement stripping. Two particularly noteworthy solutions are chemical modification, which involves adding liquid anti-stripping materials or using hydrated lime filler, and physical modification, which involves polymer modification of aggregates [16,17]. Xiao et al. conducted a comparison between two liquid anti-stripping agents and hydrated lime to determine their impact on the moisture susceptibility of WMA. They discovered that while liquid ASA additives were able to improve the indirect tensile strength of the mixtures, they exhibited a limited ability to resist moisture when compared with hydrated lime, regardless of the type of WMA and aggregate used [18]. Nazirizad et al. performed a study to examine how the addition of hydrated lime and a liquid anti-stripping agent (Iterlene In/400-S) affects the moisture susceptibilities of asphalt mixes. The results demonstrated that the asphalt mixture incorporating liquid anti-stripping additive exhibited superior resistance to water damage in comparison to the asphalt mixture incorporating hydrated lime. Shiqi et al. utilized acylimine and fatty amine anti-stripping agents in combination with rock asphalt. The study showed that rock asphalt enhanced the asphalt's resilience to deformation at high temperatures and at a broad frequency range [19]. A considerable fraction of liquid additives demonstrated enhanced unconditioned characteristics in asphalt mixtures. However, these improvements were temporary when subjected to moisture conditioning. The temporary efficacy of this led to the investigation of a new nanotechnological method, specifically the use of organo–silicon-based anti-stripping additives, which is currently gaining more attention in the sector. This cutting-edge technology utilizes the strong chemical connection found in silicon atoms, which is widely acknowledged as one of the most durable relationships in the natural world. The use of this organo–silicon framework offers a viable solution to the problems related to moisture vulnerability, potentially leading to a significant advancement in maintaining improved properties of asphalt mixtures, especially in the field of moisture conditioning. The increasing fascination with this cutting-edge nanotechnology highlights the continuous effort to develop stronger and more resistant asphalt compositions in the field of pavement materials [20–22]. Zycotherm and Zycosoil are liquid silane-based additives that effectively enhance the resistance of asphalt mixtures to moisture damage. These additives act as silane coupling agents, forming covalent bonds between the organic and inorganic phases. This results in the formation of durable adhesive bonds that remain intact even under unfavorable environmental conditions [23,24]. Arabani et al. examined the impact of Zycosoil as an anti-strip agent on the moisture sensitivity of warm mix asphalt (WMA) using surface-free energy analysis. They found that the addition of Zycosoil strengthened the adhesion between the asphalt binder and aggregate in dry and wet conditions by enhancing the surface energy [25].

Choosing the appropriate type and dose of anti-stripping additives is no doubt vital to the intended performance. Therefore, it is critically important to determine the dose of the additives used in the modification of asphalt binders. Accurate calculation of the amount of additives is crucial for effectively altering asphalt binders. Therefore, it is essential to maintain strict control and management over the quantity of additives used.

Within the domain of asphalt binder modification, difficulty emerges when confronted with siloxane-based anti-stripping liquid agents. Regrettably, currently, there is a conspicuous lack of reliable techniques for precisely quantifying the dosage of these chemicals. This inadequacy presents a substantial obstacle in the management of quality and the oversight of changed binders in the processes of quality administration (QA) and quality control (QC). The lack of a means to quantify the dosage of siloxane-based anti-stripping liquid agents not only makes it difficult to evaluate their efficacy but also hinders the overall quality assurance and quality control operations.

The repercussions of this measurement gap reverberate through the practical applications of these promising and innovative technologies. The hindered QA/QC processes impede the seamless integration of modified binders into asphalt mixture pavements, a technology that has gained increasing traction, particularly in the vast network of Georgia highways covering millions of miles. The potential benefits of improved asphalt performance, durability, and longevity are compromised when the dosage of critical additives cannot be precisely ascertained.

This study aimed to assess the possibility of measuring the amount of siloxane-based anti-stripping liquid agents, specifically Zycotherm, in modified asphalt binders using a combustion-based technique. This study comprehensively analyzed commonly utilized equipment for quantitative analysis of asphalt binders and assessed their viability in terms of repeatability, accuracy, and measurement duration. The method focused on selecting appropriate equipment, preparing samples, and using calculation formulas to obtain the utmost precision, consistency, and replicability. Ultimately, the findings from this research are anticipated to contribute significantly to the advancement of quality administration (QA) and quality control (QC) practices in the field of asphalt binder modification. Successful implementation of a combustion-based method for detecting Zycotherm dosage can potentially eliminate the existing challenges associated with the lack of effective measurement techniques. This, in turn, would pave the way for broader and more efficient utilization of siloxane-based anti-stripping liquid agents, fostering advancements in the durability and performance of asphalt mixtures.

2. Materials and Methods

2.1. Materials

2.1.1. Asphalt

In this study, PG 64-22 asphalt binder was used as a base binder, which was later modified by Zycotherm. Table 1 displays the precise technical specifications of the base asphalt binder.

Table 1. Basic properties of PG 64-22 asphalt binder.

Aging Condition	Test Property	Standard	Value	Requirement
Unaged	Viscosity @ 135 °C (cP)	AASHTO PP6	535	Maximum 3000 cP
	G*/sin δ @ 64 °C (kPa)	AASHTO PP6	1.92	Minimum 1.00 kPa
RTFO Aged	G*/sin δ @ 64 °C (kPa)	AASHTO PP6	3.26	Minimum 2.20 kPa
PAV Aged	G*/sin δ @ 25 °C (kPa)	AASHTO PP6	4695	Maximum 5000 kPa
	Stiffness @ −12 °C (MPa)	AASHTO PP6	255	Maximum 300 MPa
	m-value @ −12 °C	AASHTO PP6	0.36	Minimum 0.3

2.1.2. Zychotherm

The qualities of asphalt were improved with the use of Zycotherm, which was used as an additive. The Zycotherm product is essentially an updated version of the Zycosoil product, with its characteristics being improved in every way. Zycosoil is a chemical that derives from silanol groups (Si–OH) and is classified as an organosilane. It is possible for silanol groups to be reactive and to form a siloxane linkage (Si–O–Si) with silanol groups that are present on mineral surfaces. These silanol groups can be found on minerals that are found on the surface, in soil, and in gravel. Crucially, the hydrophobic siloxane linkages created through the reaction of silanol groups with mineral surfaces exhibit remarkable resilience. Unlike conventional methods where hydrophobic agents may be easily removed through washing, the siloxane linkages formed by Zycotherm resist such removal. This resilience is attributed to the strong chemical bonds forged between the siloxane linkages and the surface materials, ensuring a lasting and effective modification of the asphalt.

The physical and chemical characteristics of Zycotherm, which were utilized in this investigation, are outlined in Table 2.

Table 2. Basic properties of Zychotherm.

Property	Zychotherm Value
Color	Pale Yellow
Form	Liquid
Viscosity @ 25 °C	<3000 cP
Freezing Point	5–7 °C
Flash Point	>80 °C

2.1.3. Mixing Process of Zycotherm in Asphalt Binder

The wet approach utilized a low-speed mixer to integrate Zycotherm into the base binder PG 64-22. Zycotherm was added to the binder in three different ratios: 0%, 0.05%, and 0.1% by weight of the asphalt binder [26]. The mixing procedure entailed a combination of Zycotherm and asphalt binder at a low shear mixing speed of 700 rpm for a length of 45 min. The entire mixing procedure was conducted at a regulated temperature of 135 °C.

To achieve the required result, it was necessary to slowly and continuously add Zycotherm to the binder while ensuring constant stirring. The rigorous technique guarantees a comprehensive and uniform incorporation of Zycotherm, enhancing the overall efficiency of the asphalt mixture. The precision required in this wet approach is emphasized by the requirement of following exact quantities and regulated mixing circumstances. This highlights the significance of the method in optimizing the qualities of the asphalt binder to improve performance.

2.2. Test Methods

2.2.1. Oxygen Bomb Calorimeter

Experimental Apparatus

This study utilized an oxygen bomb calorimeter manufactured by PARR Instrument Company INC (shown in Figure 1). The interior chamber of the calorimeter was filled with deionized water. The temperature of the water was monitored with a high precision of 10^{-4} K at 30 s intervals using a SWC-II digital Beckmann thermometer. Furthermore, the Zychotherm mixed asphalt binder samples were compressed using a tablet machine at a laboratory size. The samples were weighed using an Ohaus PX224 Pioneer Analytical Balance, which has a minimum sensitivity of ±0.1 mg.

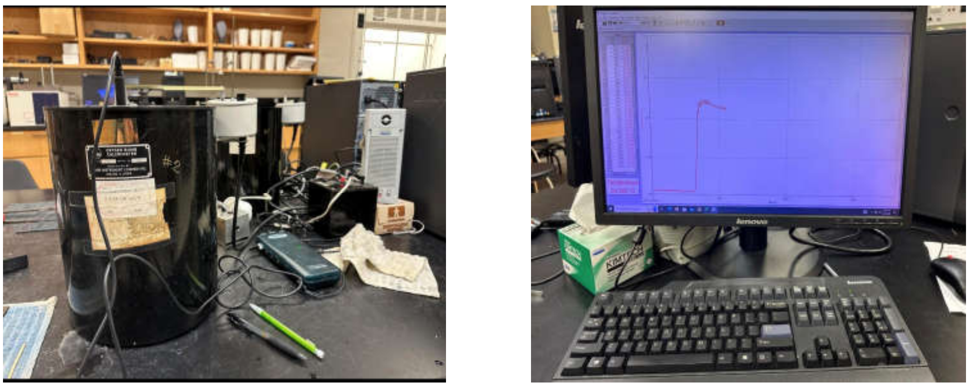


Figure 1. Oxygen calorimeter setup.

Experimental Procedure

To reliably assess the heat combustion value of binder samples, it was necessary to strictly adhere to the experimental techniques. The primary experimental protocols were presented as follows.

In the beginning, the Ohaus PX224 Pioneer Analytical Balance was utilized in order to accomplish the task of measuring the mixture of Zychotherm and asphalt binder. In the subsequent step, a tablet machine designed for use in a laboratory was utilized to compress it into slices. After that, the ignition wire was connected to the ignition electrodes, and then the cover of the oxygen bomb was attached to the bomb. After some time had passed, the lid was firmly secured, and oxygen was poured into the oxygen bomb until the gauge pressure reached the value of 4 MPa that had been set beforehand. Next, the testing circuit was connected to the other circuits. After some time had passed, approximately two kilograms of water was poured into the inner barrel, and the procedure of weight measuring started. The approach that was proposed in the relevant literature was made use of to analyze the data that was collected. One thing that should be brought to your attention is the fact that each mixed sample condition required five iterations of the measurement. This experiment follows the ASTM D4809 standard for running the test.

2.2.2. Ignition Oven

Experimental Apparatus

This study utilized two different sizes of the NCAT Asphalt Content Furnace, a product developed by Thermo Fisher Scientific firm (shown in Figure 2).

(**a**) Small ignition oven (**b**) Large ignition oven

Figure 2. Ignition oven.

Experimental Procedure

A 20 g sample was initially weighed using an Ohaus PX224 Pioneer Analytical Balance and then transferred into a rolling thin film oven container. Subsequently, the RTFO bottles were positioned within the expansive furnace oven to undergo combustion. Following the combustion procedure, charred specimens were gathered and subsequently relocated to the compact furnace for ignition. Once the ignition residue samples were gathered, they were measured using the balance. Figure 3 shows the step-by-step procedure.

For the 10 g samples, the sample was directly placed in the tiny furnace for ignition, and the residue collected after ignition was weighed.

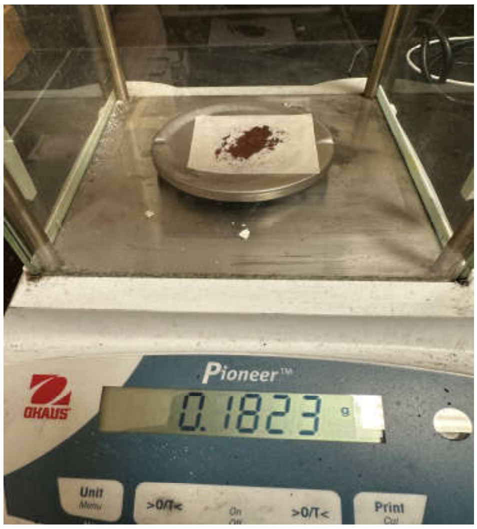
(**a**) Sample in RTFO bottle

(**b**) RTFO bottle in large oven

(**c**) Sample after burning

(**d**) Burning residue in ignition oven

(**e**) Residue after ignition



(**f**) Ignition residue collection

Figure 3. Burning and ignition of a large sample: (**a**) Weighed sample in RTFO bottle; (**b**) RTFO bottle placed in large oven for burning; (**c**) samples after burning; (**d**) burned sample placed in small oven for ignition; (**e**) residue after ignition; (**f**) ignition residue.

2.2.3. Scanning Electron Microscopy (SEM)

A scanning electron microscope (SEM) can capture images of the surfaces of a wide variety of materials in three dimensions, and these images can have a high resolution [27,28]. Although scanning electron microscopy (SEM) is most commonly used for imaging, it also has a wide range of applications in a variety of other microanalyses. In the technique known as scanning electron microscopy (SEM), a particularly concentrated electron beam is employed to irradiate a particular region for the purpose of microanalysis. The beam in question has the capability of either moving across the surface in a raster pattern or remaining stationary [29]. The microstructure characteristics of siloxane-modified asphalt were examined using the SEM EPMA (S-570) equipment developed by the Hitachi Group. This analysis aimed to observe the change and existence of silicon in asphalt binder.

2.2.4. Energy-Dispersive X-ray Spectroscopy (EDX)

Energy-dispersive X-ray spectroscopy (EDX), commonly referred to as energy-dispersive X-ray microanalysis (EDXMA), is an analytical technique used to determine the elemental composition or chemical characterization of a substance. In order for the process to be successful, there must be an interaction between an X-ray source and a sample [30]. This energy-dispersive X-ray spectroscopy (EDX) technique was used to detect the presence of silica in the residue of modified asphalt binder.

3. Results

3.1. Oxygen Bomb Calorimeter Test

For the purpose of determining the heat combustion value, a 0.5 g sample was employed for each type of sample in this investigation. A software program was utilized to generate temporal temperature data throughout the process of combustion. Figure 4 displays a temperature–time graph from which the heat combustion value was derived. In general, heat combustion is the product of the calorimeter's heat capacity and the temperature change. The change in temperature determined by the following equation.

$$\Delta T = Tc - Ta - Spre\ (b - a) - Spost\ (c - b) \tag{1}$$

where

Tc = temperature at the very highest point after combustion;
Ta = temperature at the very lowest point before combustion;
Spre = slope indicating leakage of heat "before" combustion;
Spost = slope indicating leakage of heat "after" combustion;
a = time at the lowest temperature;
c = time at the highest temperature;
b = time when 60% combustion completed.

The findings from the oxygen bomb calorimeter, which are depicted in Figure 5, indicate that the incorporation of Zychotherm additives typically resulted in a reduction in the heat combustion value. Providing an explanation for the drop in heat combustion value is something that can be done. There is a transformation of siloxanes into silicon dioxide (SiO_2), as well as the creation of volatile carbon dioxide and water when siloxane-based additives are subjected to oxidation in oxygen [31,32]. The following is an example of the oxidation reaction of materials containing siloxane:

$$C_8Si_4H_{24}O_4 + 16O_2 \rightarrow 4SiO_2 + 8CO_2 + 12H_2O$$

$$C_{10}Si_5H_{30}O_5 + 20O_2 \rightarrow 5SiO_2 + 10CO_2 + 15H_2O$$

Upon analyzing the five repetitions for each sample of heat combustion, it is evident that the heat combustion value of the 0.05% modified and 0.1% modified binder fell by 1.18% and 1.61%, respectively, compared with the basic binder, resulting in the lowest heat combustion value. For optimal combustion efficiency, these values decreased by 1.34% and 1.72%, correspondingly. The standard deviations of the five repetitive samples were 110.02, 61.63, and 76.31 for the unmodified and 0.05% and 0.1% for the Zychotherm-modified samples, respectively.

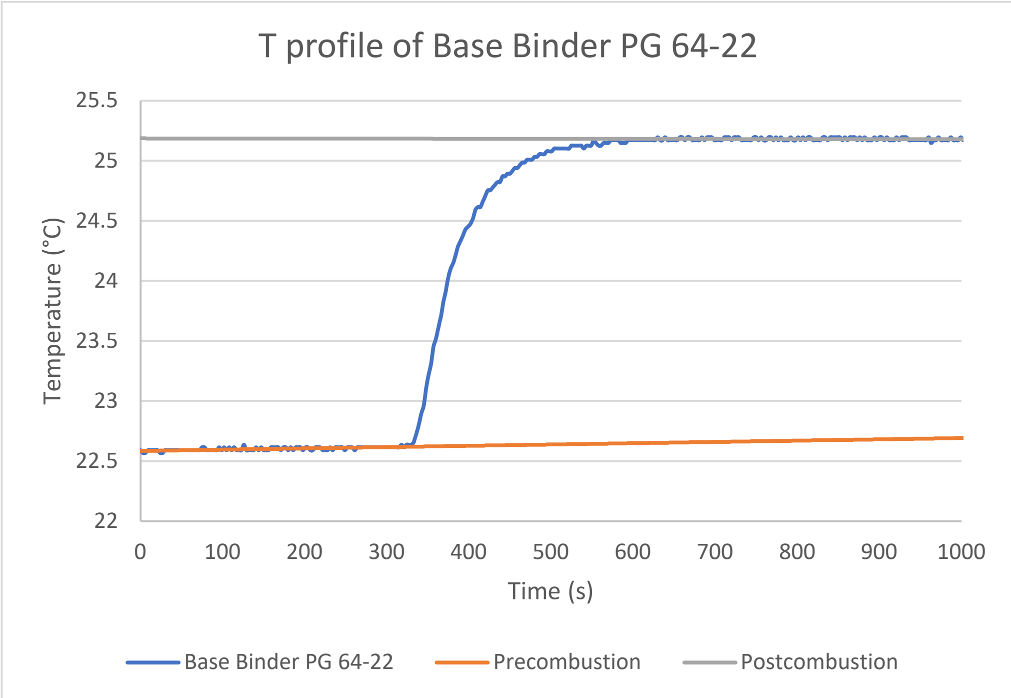


Figure 4. Temperature profile (reproduced).

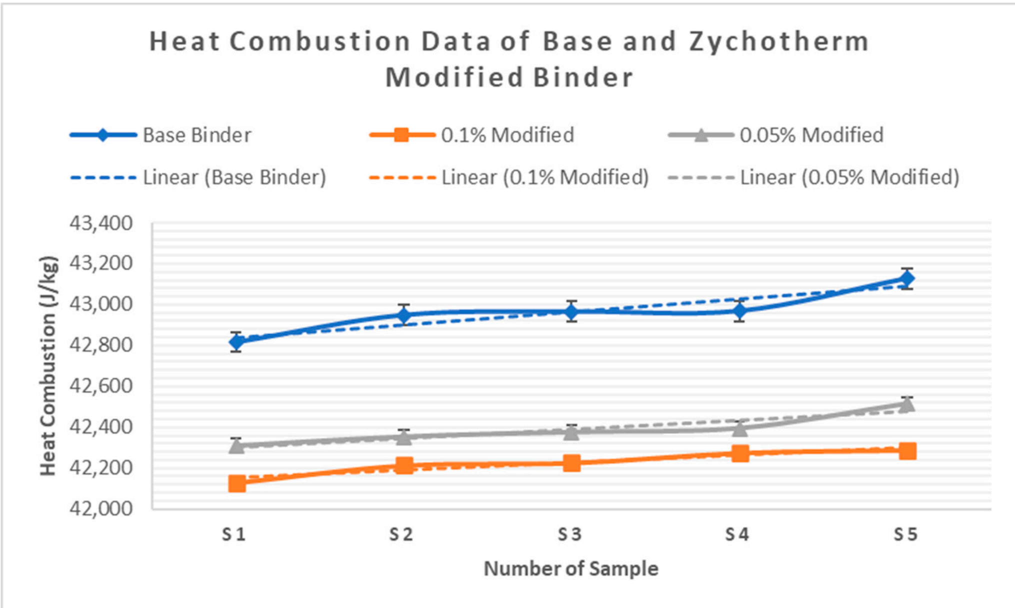


Figure 5. Heat combustion result of base binder and Zychotherm-modified binder (five repetitions for each sample) (reproduced).

The heat combustion values differed from 42,816–43,126 J/g, 42,130–42,286 J/g, and 42,310–42,514 J/g, respectively, for the unmodified, 0.05% modified, and 0.1% modified asphalt binders. We believe that the less flammable siloxanes added to the flammable asphalt matrix can retard the oxidation process and, thus, result in lower combustion heat values. Figure 6. showcases the average heat combustion value of the five repeated samples for each batch type of sample.

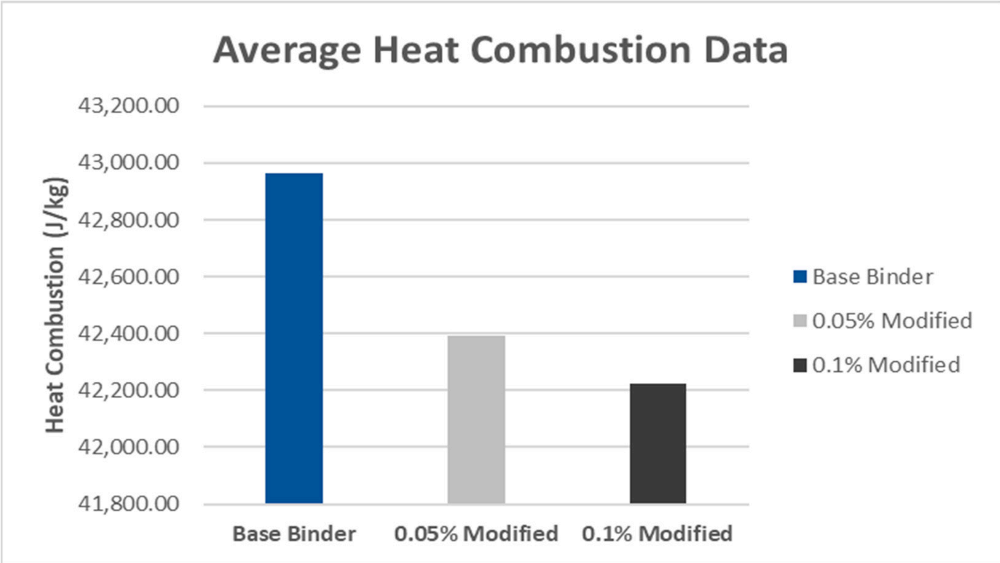


Figure 6. Average heat combustion results of base binders and Zychotherm-modified binders (reproduced).

3.2. Detection by Residue Visualization

Zychotherm can be readily identified by observing the residue left behind following the ignition of the samples. The residue of Zychotherm-modified asphalt was yellowish, while the residue of untreated binder appeared as a dark black color. This phenomenon can be elucidated. Despite being colorless, silica exhibited color in the residue. The primary factor is that the majority of asphalt binders consist of vanadium oxide (V_2O_5), which imparts a dark brown hue. Additionally, the presence of silica mixed with vanadium oxide (V_2O_5) results in a yellowish appearance in samples containing siloxanes. Figure 7 displays the color of both unmodified and modified residues. From Figure 7, it is clearly observed that the modified sample (the right one for both Figure 7a,b) showed yellowish substances, while the unmodified sample left a dark residue.

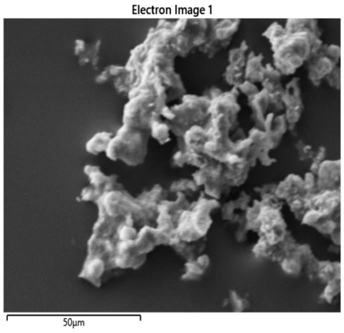
(**a**) Residue of 20 g sample

(**b**) Residue of 10 g sample

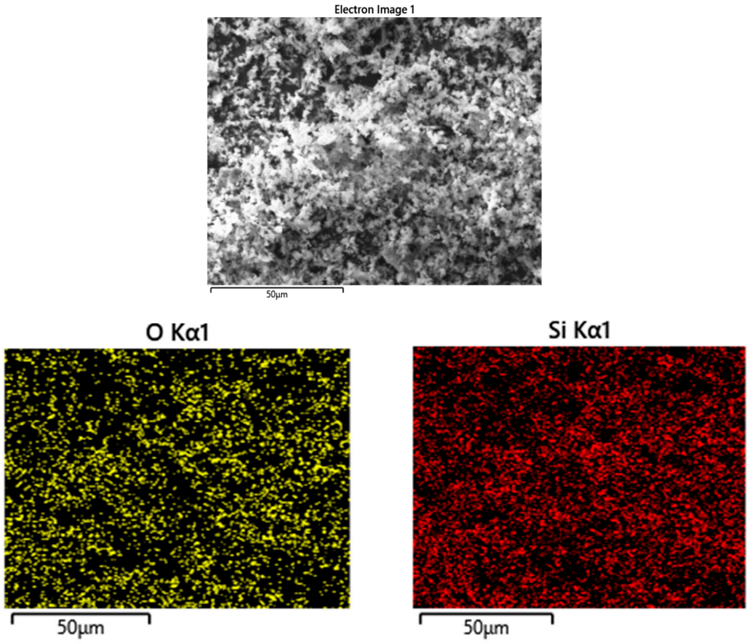
Figure 7. Residue after burning and ignition. In both figures, the left one is an unmodified sample, and the right one is a Zychotherm-modified sample.

3.3. Scanning Electron Microscopy (SEM) Analysis

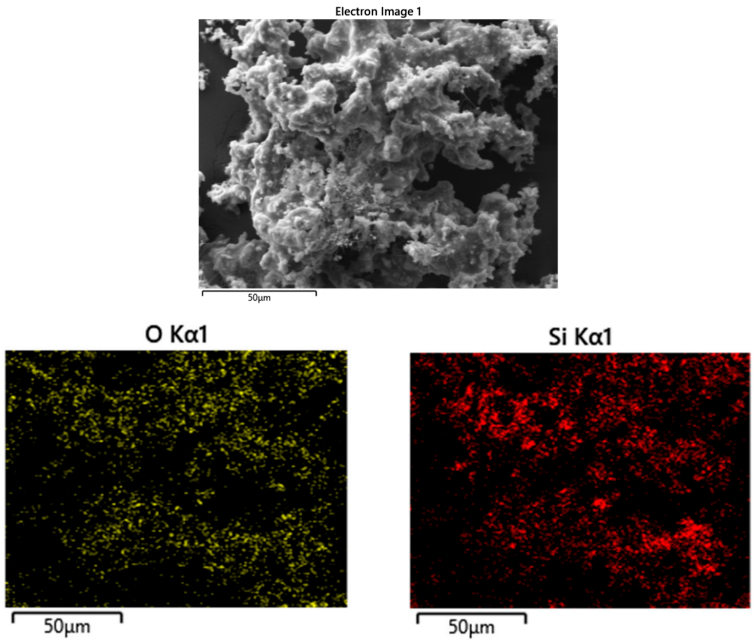
Figure 8 shows the SEM images of the modified and unmodified asphalt binders.



(**a**) Unmodified



(**b**) 0.05% modified



(**c**) 0.1% modified

Figure 8. SEM images of unmodified and Zychotherm-modified asphalt binders.

All the above images were collected using a field-emission scanning electron microscope attached to an EDX analyzer. Figure 8b,c has a medium concentration of siloxanes; Figure 8a has a low or zero concentration of siloxanes. The SEM images and EDX elemental maps shown in Figure 8 reveal that there are very few silica nanoparticles in the unmodified asphalt binder after being oxidized in air. In contrast, the number of particles significantly increased when the concentration of additives was increased, suggesting that the mass of silica particles can be employed to the quantity the additives, Zychotherm, due to the oxidation reactions discussed above.

3.4. Energy-Dispersive X-ray Spectroscopy (EDX) Analysis

Figure 9 shows the EDX analysis of the 0.1% Zychitherm-modified asphalt binder after being oxidized in the air. This binder contained a strong amount of silicon and oxygen peaks. The carbon peak came from the conductive tape used for the SEM imaging; the Al peak originated from the sample holder; the Pd came from the sputter coating. The peak of Si is proof of the asphalt binder containing silica-based Zychotherm.

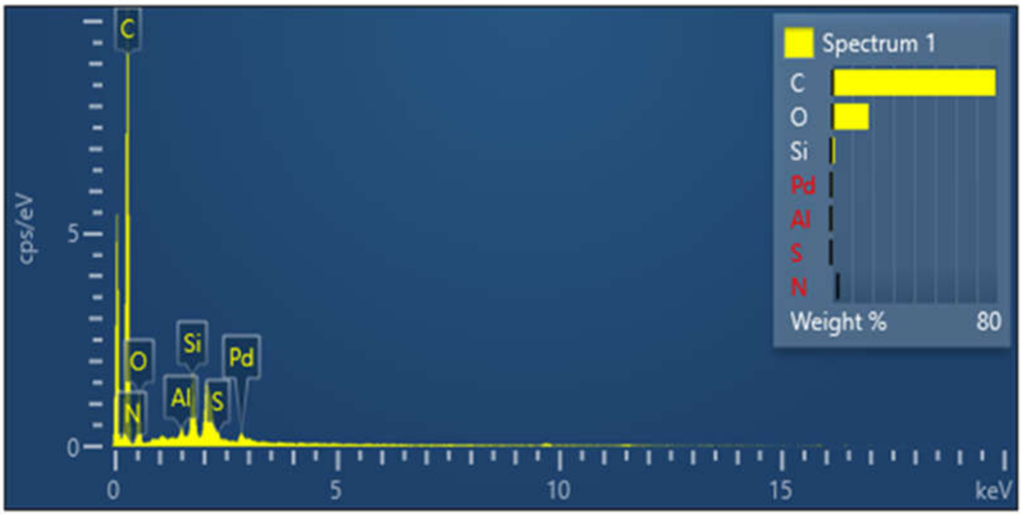


Figure 9. EDX spectrum of 0.1% Zychotherm-modified asphalt binder.

3.5. Sample Size Effect

Although it is possible to detect Zychotherm-modified asphalt binder by visualizing their residue color, it is not possible to quantify the amount of Zychotherm.

The residue from each sample was precisely weighed using an Ohaus PX224 Pioneer Analytical Balance to perform a quantitative analysis of the amount of Zychotherm. Figure 10 illustrates the difference between the actual residue weight and the theoretical residue weight for the sample totaling 20 g. When compared with the theoretical residue weight, the actual residue weights were 27% and 31% lower for samples that were treated with 0.05% Zychotherm and 0.1% Zychotherm, respectively.

The significant inaccuracy observed in this investigation is related to the volumetric expansion of the 20 g asphalt binder sample during the combustion process within the big oven. An important outcome of this extension was the spillover of a certain portion of the sample from the rolling thin film oven (RTFO) bottle. This incident sparked concerns over the possible loss of the Zycotherm component, which could undermine the precision of the testing results.

To address this difficulty, a strategic alteration was implemented in the next experimental phase to reduce the possibility of sample loss caused by volume increase. More precisely, the weight of the sample was decreased from 20 g to 10 g. The intentional modification was intended to restrict the enlargement of the asphalt binder while it was being heated, thereby avoiding any overflow from the RTFO bottles. The careful modification in the experimental procedure demonstrates a diligent attempt to acknowledge and correct the discovered difficulties, guaranteeing the protection of the sample and the related Zycotherm element.

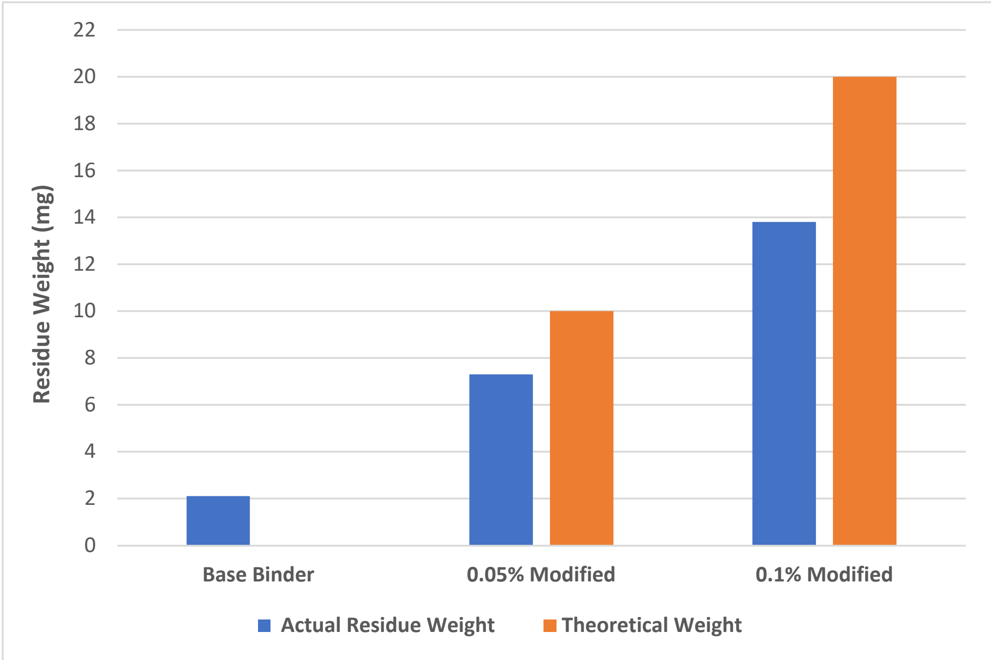


Figure 10. Residue weight of 20 g base binders and Zychotherm-modified binders.

With the weight of the sample being decreased, the precession of the results was enhanced. The gap between the actual and theoretical residue weights was significantly smaller than the 20 g sample, as observed in Figure 11, which illustrates the residue weight for both modified and unmodified binders. There was a 16% and 23% difference between the predicted residue weight and the actual residue weight for samples that were treated with 0.05% Zychotherm and 0.1% Zychotherm, respectively. Comparing the 10 g sample to the 20 g sample, the error rate for the 10 g sample was lowered to 11% and 8%, respectively.

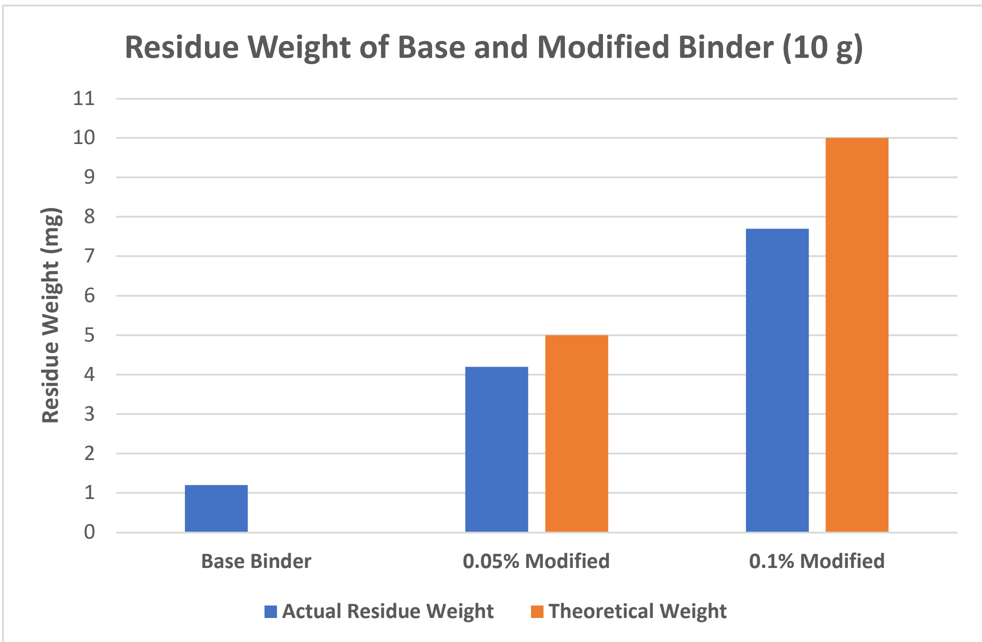


Figure 11. Residue weight of 10 g base binders and Zychotherm-modified binders.

4. Conclusions

The purpose of this research was to investigate whether it would be possible to create a detection method that would be able to measure the amount of siloxane-based anti-stripping liquid agents, more especially Zycotherm, that are present in modified asphalt binders. Following are some conclusions that can be drawn from the various results of the tests that were conducted:

1. The study makes a feasible combustion-based detection method for quantifying siloxane-based anti-stripping agent (Zycotherm) concentrations in modified asphalt binders. This method, leveraging heat combustion and ignition processes, demonstrated the capability to closely measure low doses (0.05% and 0.1%) of Zycotherm, showcasing its potential for practical application in quality control and quality assurance processes in asphalt binder modification.
2. Incorporation of Zycotherm into asphalt binders resulted in a measurable decrease in heat combustion values, with reductions of 1.34% and 1.72% for 0.05% and 0.1% Zycotherm-modified binders, respectively. This finding indicates that Zycotherm, even in minimal concentrations, can significantly affect the combustion characteristics of asphalt binders, suggesting a chemical interaction that alters the binder's thermal properties.
3. The study revealed that Zycotherm-modified asphalt binders leave a distinguishable yellowish residue after burning and ignition processes, contrasting with the dark black residue of unmodified binders. This observation not only supports the method's effectiveness in detecting Zycotherm presence but also offers a simple, visual means of preliminary assessment before quantitative analysis.
4. A direct, nearly proportional relationship was established between the amount of Zycotherm and the mass of silica residue. This correlation underscores the principle that the quantity of silica residue resulting from the combustion of siloxane-based additives can serve as a reliable indicator of the Zycotherm content in modified asphalt binders.

Even though this study made an effort to obtain the most accurate results possible, there is still room for improvement in terms of employing more sophisticated methods. Gel permeation chromatography (GPC) and Fourier-transform infrared spectroscopy (FTIR) techniques can be utilized in conjunction with one another as a combined microanalysis tool to achieve superior outcomes in subsequent research.

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