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EXPLORING MICROSTRUCTURAL CHARACTERISTICS OF ASPHALTIC CONCRETE ENHANCED WITH RECYCLED CERAMIC TILE WASTE

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Abstract
This study investigates the microstructural characteristics of asphaltic concrete incorporating recycled ceramic tile waste (CTW). Through a series of analytical techniques, including Atomic Absorption Spectroscopy (AAS), Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and Energy-Dispersive X-Ray Spectroscopy (EDX), the microstructure of CTW modified asphalt specimens was examined. Results from AAS revealed varying concentrations of metallic elements, with aluminum exhibiting the highest concentration in the control specimen, attributed to the alumino-silicate content in granite. FTIR analysis identified characteristic vibrational modes of sample molecules, indicating the presence of specific minerals in the CTW modified asphalt specimens. SEM micrographs depicted changes in structure with increasing CTW content, including the presence of cracks and increased surface area. EDX analysis identified major elements present in the specimens, with notable variations across different CTW compositions. Overall, this study contributes to understanding the microstructural changes induced by CTW incorporation in asphaltic concrete, providing valuable insights for the utilization of sustainable materials in infrastructure development.

Keyword: Asphaltic concrete, ceramic tile waste, fourier transform, spectroscopy, electron microscopy, alumino-silicate

Introduction
In Nigeria, asphalt concrete pavement serves as a pivotal component of road infrastructure, facilitating the transportation of goods and services. The improvement of asphalt properties has garnered considerable attention, prompting investigations into additives that can enhance performance. Asphalt concrete comprises coarse aggregates, fine aggregates, fillers, and bitumen as the binder, collectively influencing its overall performance.

Hot Mix Asphalt (HMA), a widely employed road construction material globally, typically consists of approximately 5% bitumen and 95% aggregate materials by weight (Liu *et al.*, 2019). Bitumen, derived from petroleum refining, acts as the crucial binder in asphaltic concrete. However, the rising production costs of HMA, attributed to diminishing petroleum resources and high-quality aggregate, necessitate exploration of alternative materials.

In response to the depletion of natural aggregates and associated environmental concerns, the utilization of recycled ceramic tile waste emerges as a promising solution. This waste, obtained from the ceramics industry or construction and demolition activities, offers both abundance and potential environmental benefits (Juan *et al.*, 2010). It not only reduces reliance on natural resources but also presents energy-saving advantages through the recovery of previously incorporated energy during production.

Several studies have investigated the feasibility of utilizing recycled ceramic tile waste as a substitute for conventional aggregate in concrete and road pavement construction, demonstrating its potential for both bituminous and concrete pavements (Silva *et al.*, 2010). Therefore, this research aims to address the scarcity and high cost of conventional aggregates in asphalt pavement production by exploring the microstructural characteristics of asphaltic concrete enhanced with recycled ceramic tile waste.

Amidst ongoing modernization and infrastructure development, the increasing demand for construction materials poses challenges in resource depletion and environmental degradation. In response, researchers are investigating alternative materials, such as recycled ceramic tile waste, to enhance asphaltic concrete performance while promoting sustainability (Ikponmwosa and Ehikhuenmen, 2017). Thus, this study focuses on exploring the microstructural characteristics of asphaltic concrete enhanced with recycled ceramic tile waste to contribute to sustainable infrastructure development.

Material

Crushed granite samples were sourced from a local quarry in Osogbo, Osun State, and underwent thorough examination for physical and micro-mechanical properties. Prior to gradation, the samples were dried for 24 hours to ensure consistency. Crushed ceramic stone was obtained through the disintegration of quarry rock, boulders, cobbles, or sizable gravel.

Fine aggregate, comprising natural sand or crushed stone not exceeding 5 mm in size, was sourced from natural aggregate deposits along Iwo Oshogbo road. The fine aggregate sample underwent washing, followed by a 24-hour drying period and grading. Various tests, including sieve analysis, moisture content, specific gravity, rate of water absorption, aggregate abrasion, crushing, and impact values, were conducted to assess its properties.

Grade 60/70 bitumen was used in the production of the asphaltic mixtures incorporating crushed ceramic tile waste. Sourced from the Ogun State bitumen plant, the bitumen underwent rigorous testing for penetration, flash point, ductility, and softening point to ensure compliance with quality standards.

The experimental procedures for CTW modified asphalt involved the preparation of six different mixtures varying in the percentage of crushed tiles and granite, along with straight run bitumen. The mixtures ranged from 100% granite and sand with no crushed tiles to 100% crushed tiles and sand with no granite. Table 1 outlines the composition of the mixtures and their representation in the result analysis.

Table 1: Matrix and Indications

MATRIX	SAMPLE
100% Granite, 0% Crushed Tiles	A
75% Granite, 25% Crushed Tiles	B
50% Granite, 50% Crushed Tiles	C
25% Granite, 75% Crushed Tiles	D
0% Granite, 100% Crushed Tiles	E

Methods

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Atomic Absorption Spectroscopy (AAS)

Atomic Absorption Spectroscopy (AAS) was employed to determine the concentrations of metallic elements in the CTW modified asphalt specimens. This analytical technique utilizes electromagnetic wavelengths emitted by a light source, with different elements absorbing these wavelengths uniquely. The samples were pulverized into dust, aspirated, and aerosolized before being mixed with explosive gases such as acetylene and air or acetylene and nitrous oxide. Subsequently, they were burned in a flame to release individual atoms. The analysis was conducted at the chemistry laboratory of Redeemer's University, Ede, Osun State, using approximately 200 grams of each matrix of the CTW modified asphalt specimen. The Photomultiplier tube detector provided a wide range sensitivity for atomic absorption analysis.

Mineral Determination: Mineral contents were determined using an atomic absorption spectrophotometer (AAS) following AOAC (2003) methods. Spectrophotometer UV5, offering rapid full-spectrum scanning, was utilized. A Shimadzu Atomic Absorption Spectrophotometer model AA-6800 was employed to determine the concentrations of Iron (Fe), Aluminium (Al), Lead (Pb), Copper (Cu), and Cadmium (Cd).

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was conducted to determine the concentration of specific minerals in the CTW modified asphalt specimens. This technique utilizes electromagnetic wavelengths to produce an infrared spectrum, identifying organic and inorganic compounds. The SHIMADZU FTIR8400S instrument was used at Redeemer's University, Ede, Osun State. KBr pellets were prepared and scanned as background, and then each matrix of the CTW modified specimen was mixed with KBr, pelletized, and scanned in transmittance mode.

Scanning Electron Microscopy (SEM) Energy-Dispersive X-Ray Spectroscopy (EDX)

Scanning Electron Microscopy (SEM) and Energy-Dispersive X-Ray Spectroscopy (EDX) were utilized to provide topographical and elemental information of the CTW modified asphalt specimens. The Phenom Pro X Model: 800-07334 instrument was used, operating at a voltage of 15KV. FESEM was employed for clearer, higher resolution images. EDX analysis was conducted to identify the elemental composition by firing an electron beam at the sample and measuring the resulting X-ray energy.

Results

Atomic Absorption Spectroscopy

The findings from Atomic Absorption Spectroscopy (AAS) are detailed in Table 2 and Table 3. Notably, the control specimen (sample A) exhibited the highest concentration of aluminum at 16.2 mg/l, attributed to the high alumino-silicate content in granite, which was abundant in this sample. Sample A also demonstrated the highest lead concentration at 37.1 mg/l, with samples C and E closely following at 34.5 mg/l each. Sample E showed the highest ferrous content at 11.05 mg/l, while samples B and C recorded the highest copper concentration at 12.95 mg/l each. Sample E displayed the highest cadmium concentration at 18.4 mg/l.

Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) was utilized to analyze the vibrational modes of the sample molecules, revealing distinct bands indicative of various elements. The spectrum showed major bands at approximately 3464 cm^{-1} for aluminum in specimen D, 534 cm^{-1} for lead in specimens C and E, 459 cm^{-1} for iron in specimens B, C, D, and E, and 1456 cm^{-1} for cadmium in all specimens, as presented in Table 4.6. Peaks between 3464 cm^{-1} and 3250 cm^{-1} suggested the presence of hydrogen bonds, including amino, hydrate, and hydroxyl compounds. The O-H stretching band for the molecular structure of the CTW matrix ranged from 3464 cm^{-1} to 3250 cm^{-1}, indicating decomposition of $Ca(OH)_2$. These findings align

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with established peak values specified by Nandiyanto *et al.* (2022), confirming the suitability of the obtained values for asphalt concrete.

Table 2: Average quantities of heavy metals in CTW Modified Asphalt specimen produced (mg/l) Atomic absorption spectroscopy (AAS) results

SPECIMEN	Aluminim	Lead	Ferrous	Copper	Cadium
A	16.2	37.1	9.5	14.4	16.4
B	10.7	28.6	10.2	12.95	12.7
C	11.6	34.5	8.7	12.95	17.75
D	10.05	27.6	9.8	11.8	13
E	12.5	34.5	11.05	13.5	18.4

Table 3: Average concentration of heavy metals in CTW Modified Asphalt specimens produced (cm-1) Fourier transform infrared analysis (FTIR)

Specimen	Aluminum	Lead	Ferrous	Copper	Cadmium
A	3423.76	426.21	457.14	3464.27	1456.3
B	3458.48	459.07	459.07	3464.27	1456.3
C	3441.12	534.3	459.07	3464.27	1456.3
D	3464.27	418.57	459.07	3464.27	1456.3
E	3423.76	534.3	459.07	3464.27	1456.3

Table 4: Elements and Number in the CTW Modified Asphalt Specimens

Specimens	Size of Pores (µm)	Oxygen		Carbon		Silicon		Aluminium	
		Atomic Conc.	Wt Conc.	Atomic Conc.	Wt Conc.	Atomic Conc.	Wt Conc.	Atomic Conc.	Wt Conc.
0% tiles (A)	537	63.43	52.02	15.18	9.35	14.10	20.30	5.45	7.54
25% tiles (B)	537	64.11	59.33	9.80	6.81	11.69	18.99	4.28	6.67
50% tiles (C)	537	66.53	55.33	11.14	7.64	11.54	18.52	3.86	5.96
75% tiles (D)	537	62.27	53.41	11.19	7.21	11.78	17.74	4.95	6.57
100% tiles (E)	541	62.11	58.41	21.88	15.45	11.62	19.17	4.40	6.97

Scanning Electron Micro-scopy and Energy Dispersion X-Ray Spectro-scopy

The microstructural characteristics of the CTW modified asphalt specimens were examined utilizing scanning electron microscopy (SEM), as depicted in Figure 4(a-e) at a magnification of 500x. Sample A exhibited bulky structures with fine particulate aggregates. As the CTW content increased (ranging from 25% to 100%), more cracks were observed in the samples, indicating the potential for increased total air voids. This observation aligned with the results obtained from water absorption capacity measurements. Additionally, an increase in CTW content led to an expansion of the surface area of the structures, suggesting enhanced permeability, consistent with permeability measurements obtained from the permeameter.

Moreover, SEM analysis revealed more flocculated structures at higher CTW content, known to contribute to increased permeability. Samples with 100% granite exhibited the least permeability and void content, correlating with their optimal Indirect Tensile Strength.

Energy dispersive X-ray spectroscopic (EDX) studies of the CTW modified asphalt specimens were conducted, and EDX spectra corresponding to SEM images of each sample were evaluated. The spectra identified four major elements: Oxygen, Carbon, Silicon, and Aluminium, essential constituents of asphaltic concrete. Other detected elements were

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attributed to common earth metals or non-metals. Specimen C exhibited a high proportion of atomic concentration of Oxygen, Specimen A had the highest percentage of atomic concentration of Carbon, and Specimen E showed the highest percentage of atomic concentration of Silicon and Aluminium. The high Silicon and Aluminium contents in Specimen E likely contributed to its elevated coefficient of permeability.

Furthermore, comparing the Flow and Stability values of Specimen E with the recommended values for medium traffic, it was found that Specimen E met the requirements. This observation was corroborated by SEM analysis, which indicated a higher pore value in micrometers for Specimen E.

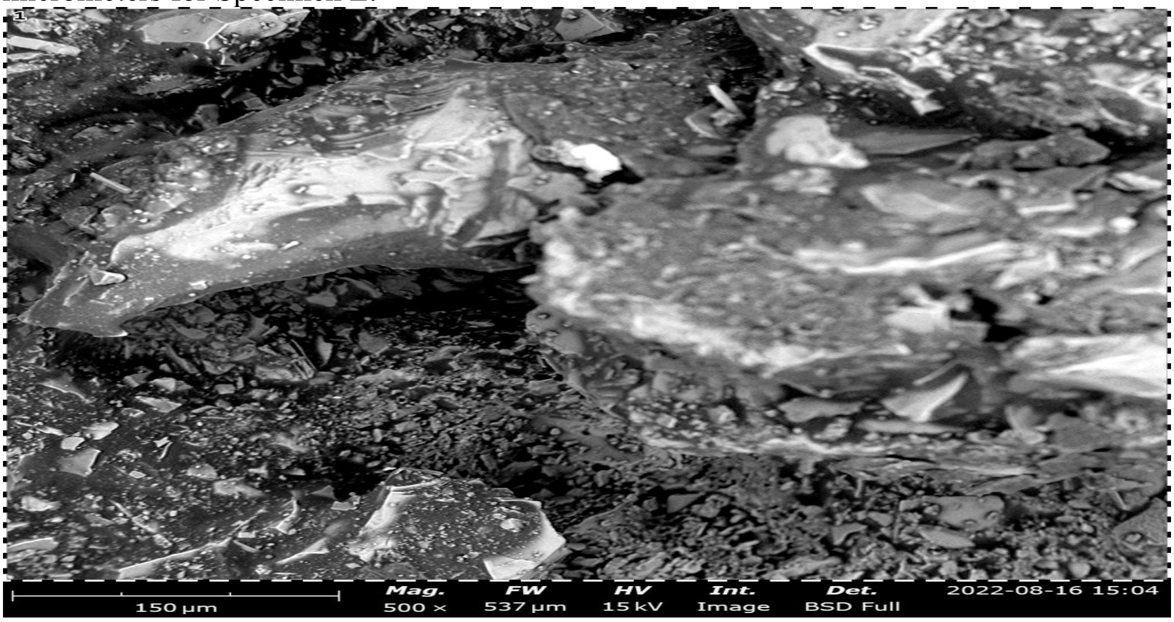

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Figure 1 (a) SEM Image for Specimen A

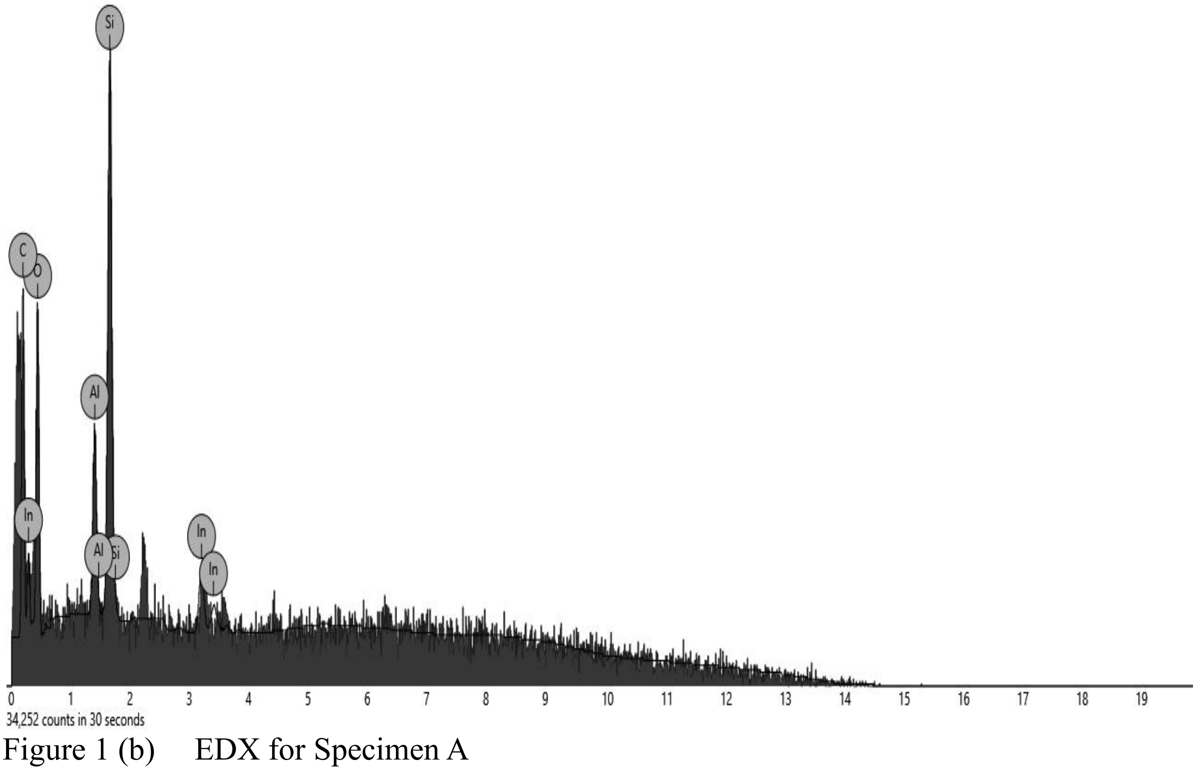

Figure 1 (b) EDX for Specimen A

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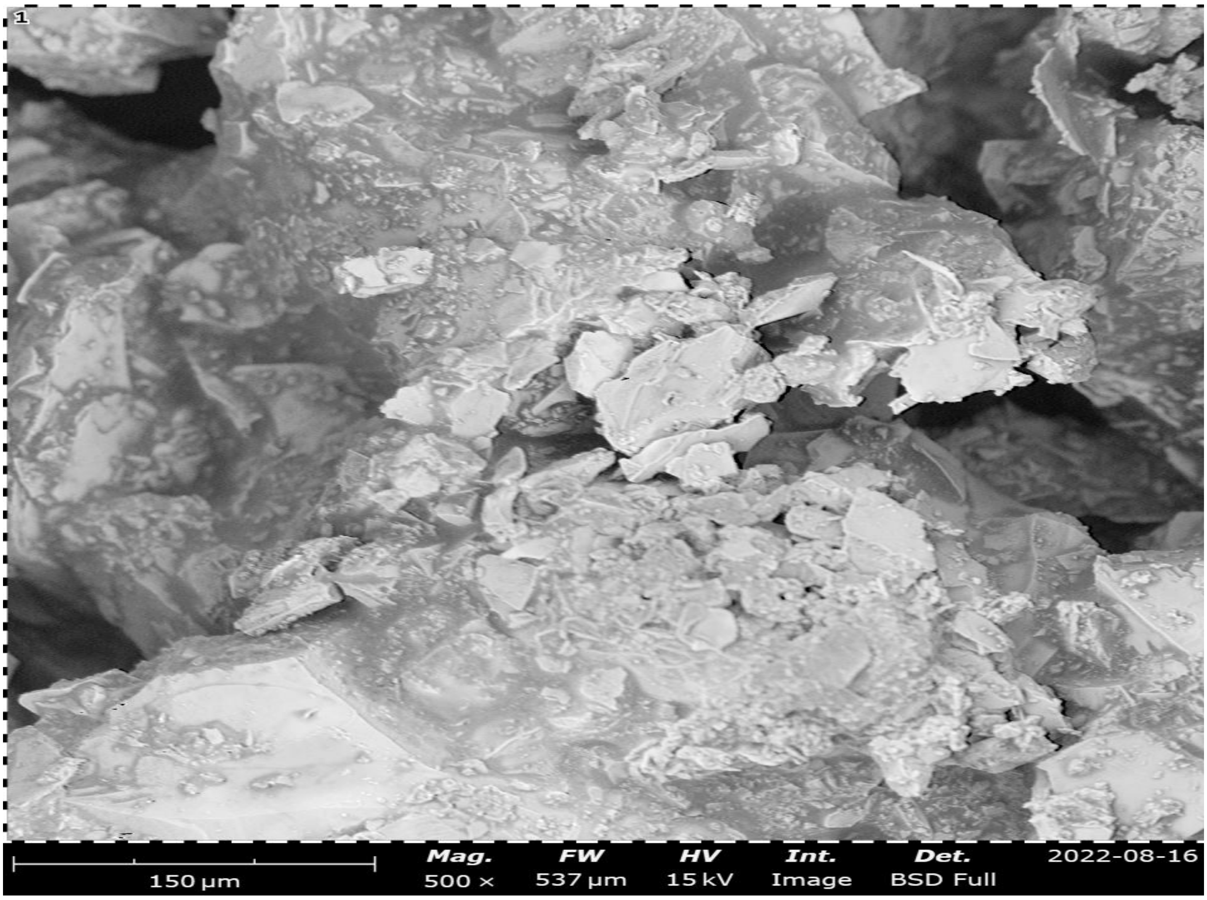

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Figure 2 (a) SEM Image for Specimen B

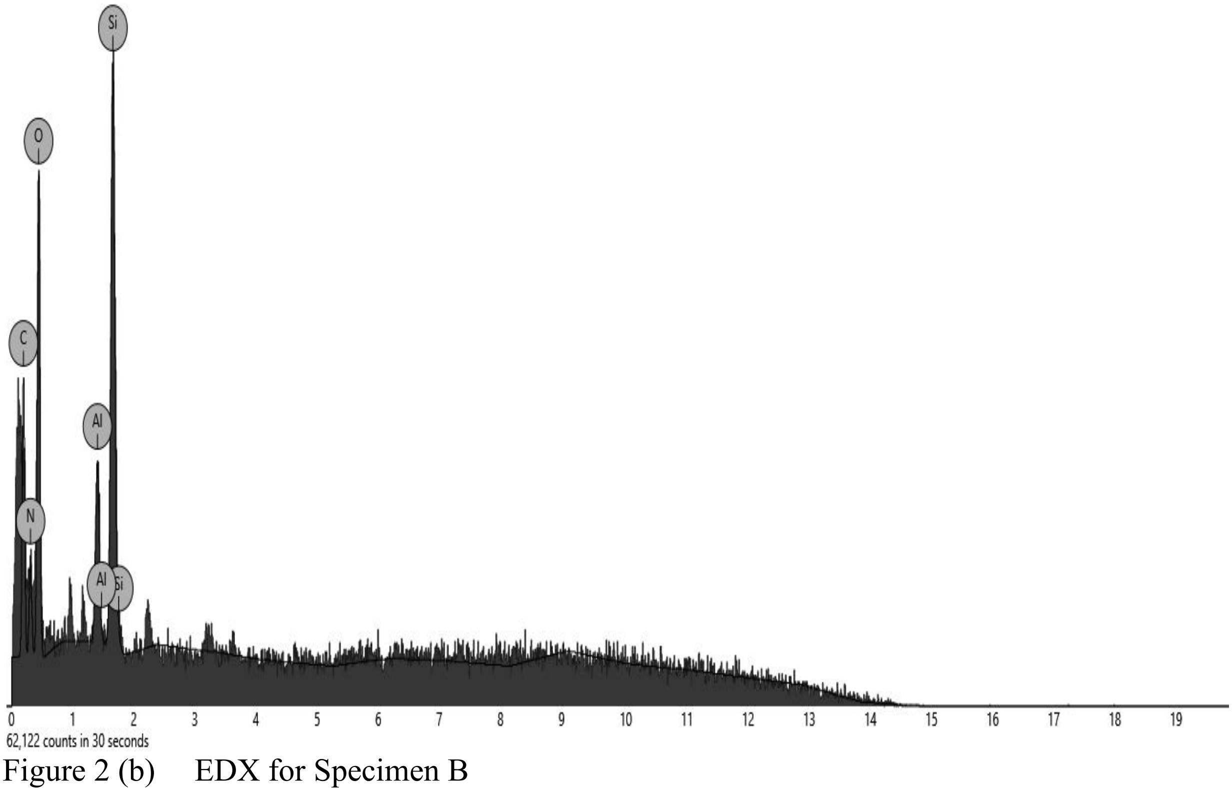

Figure 2 (b) EDX for Specimen B

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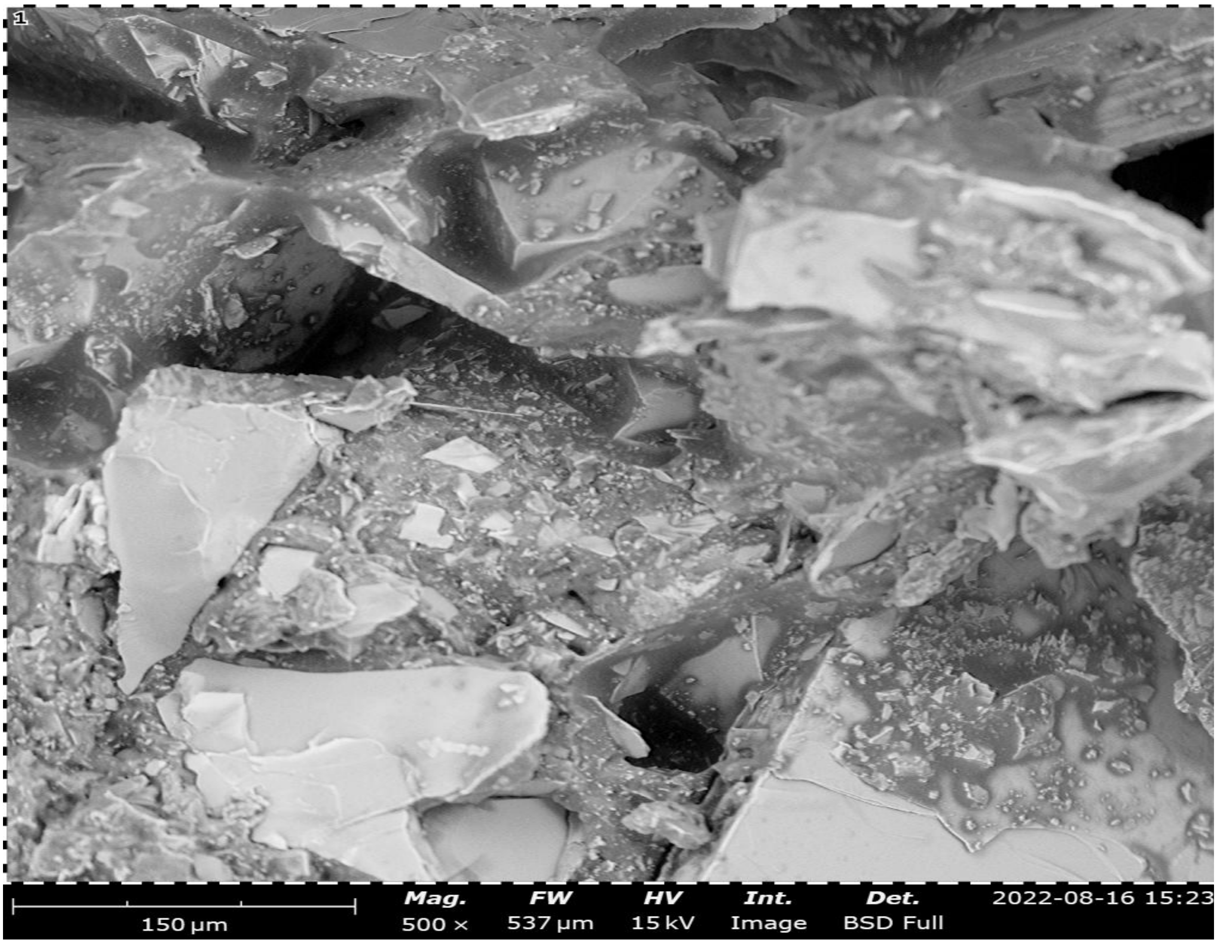

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Figure 3 (a) SEM Image for Specimen C

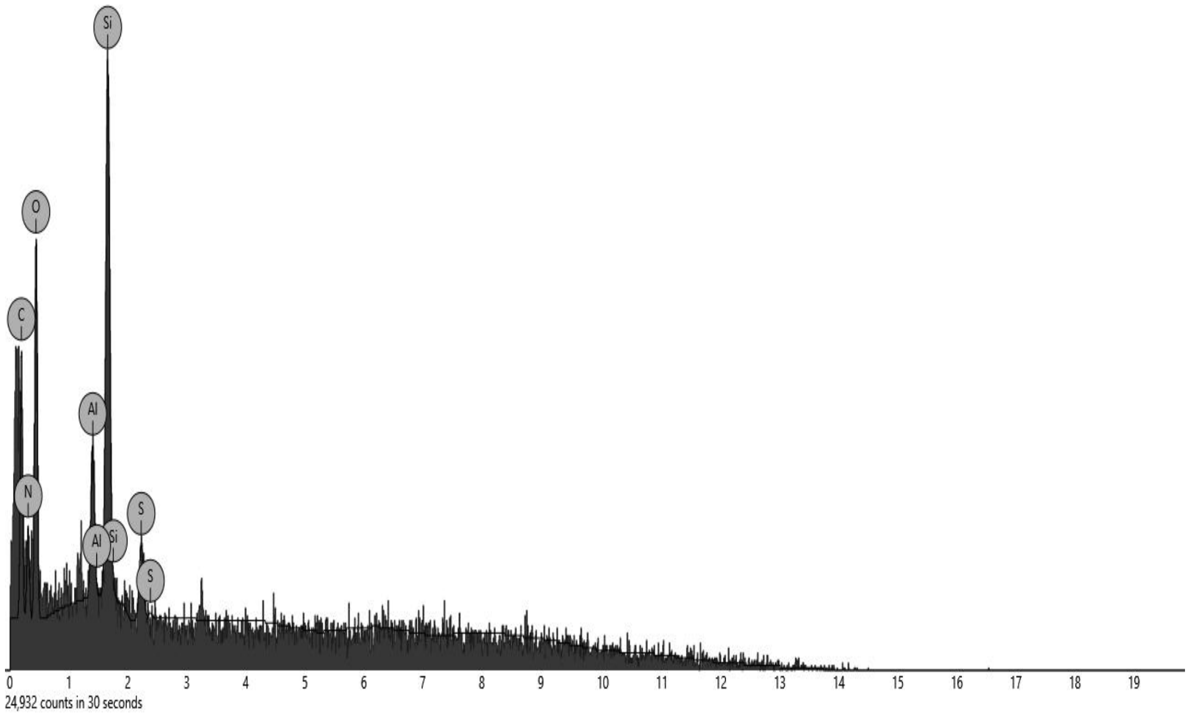

Figure 3 (b) EDX for Specimen C

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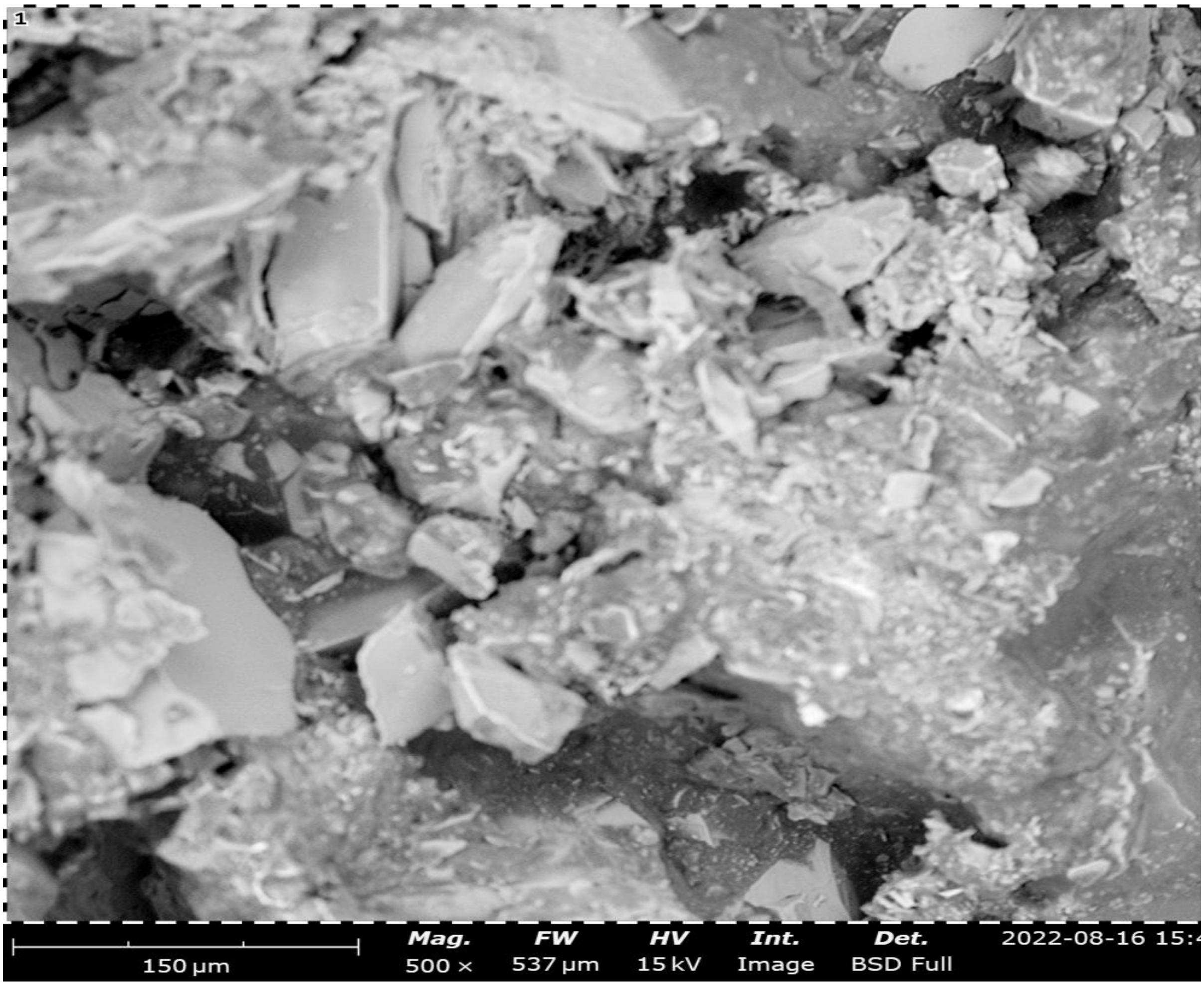

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Figure 4 (a) SEM Image for Specimen D

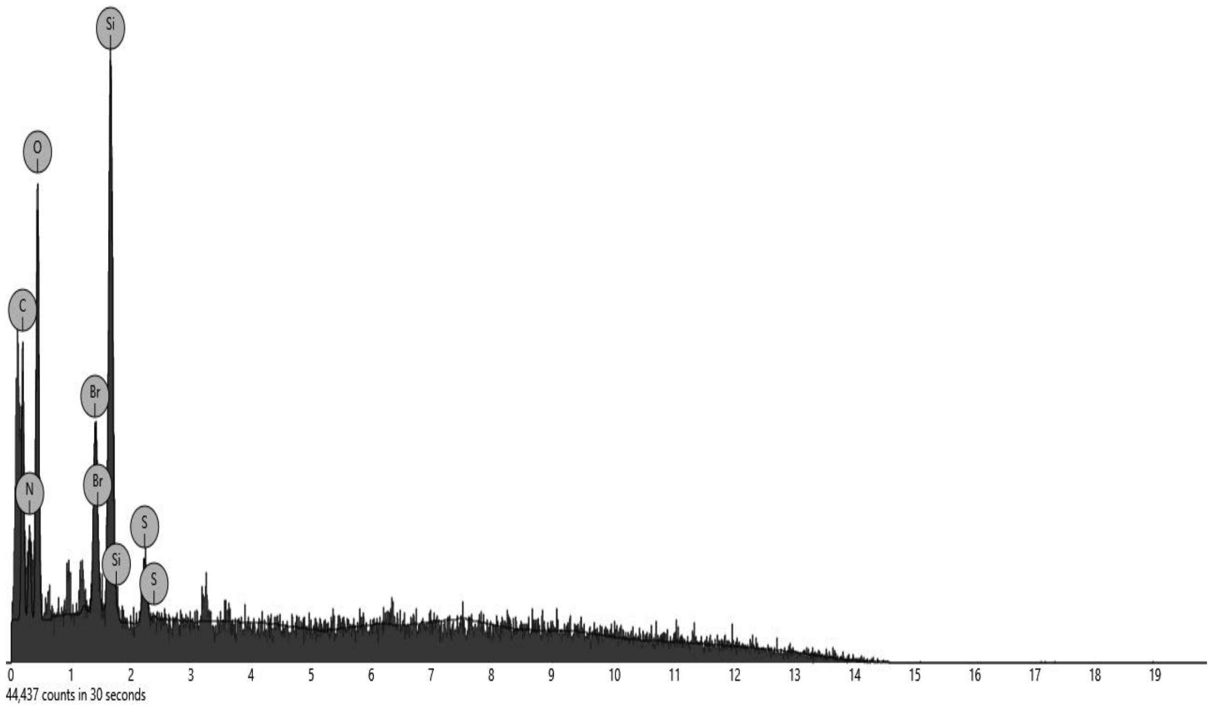

Figure 4 (b) EDX for Specimen D

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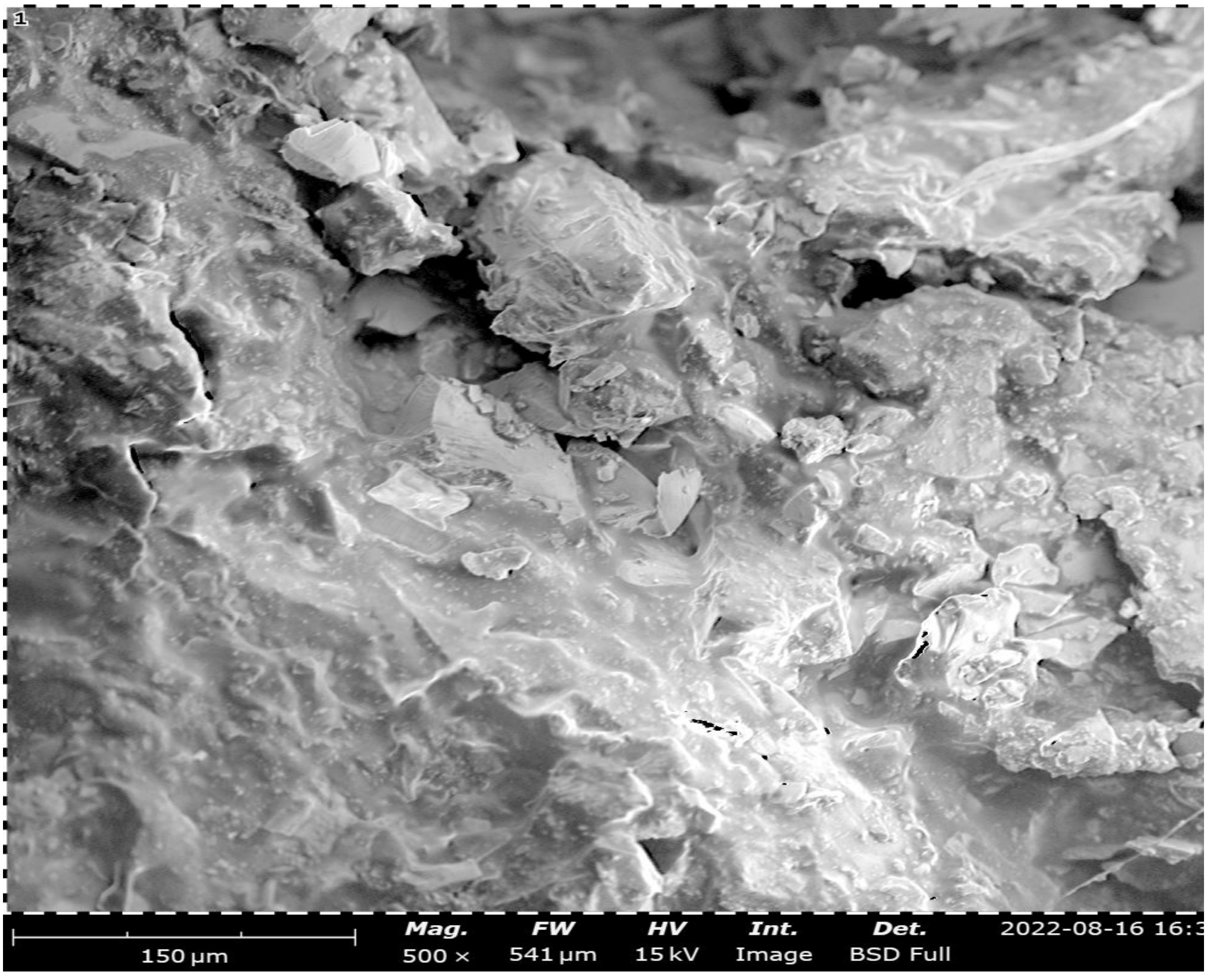

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Figure 5 (a) SEM Image for Specimen E

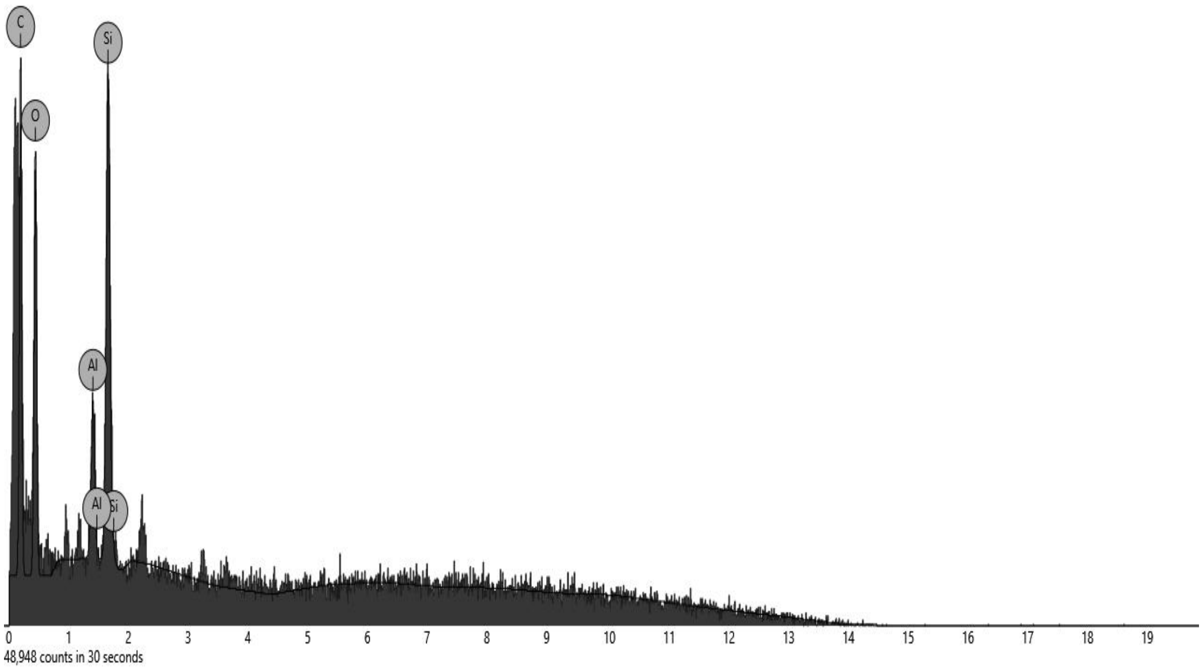

Figure 5 (e) EDX for Specimen E

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Conclusion

In conclusion, this study delved into the microstructural analysis of asphaltic concrete enhanced with recycled ceramic tile waste (CTW). Through a comprehensive examination utilizing various analytical techniques, including Atomic Absorption Spectroscopy (AAS), Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and Energy-Dispersive X-Ray Spectroscopy (EDX), valuable insights were gained into the properties and characteristics of the CTW modified asphalt specimens.

The results revealed that the incorporation of CTW influenced the microstructure of the asphaltic concrete, leading to notable changes in properties such as permeability, void content, and indirect tensile strength. SEM images depicted an increase in cracks and surface area with higher CTW content, indicating the potential for enhanced permeability but also suggesting a compromise in structural integrity. Moreover, EDX analysis identified key elements present in the specimens, with notable variations observed across different CTW compositions.

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