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Microstructural Characteristics of Fly Ash Geopolymer Modified Asphalt Binder

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Abstract. The incorporation of by-product materials, such as fly ash geopolymer, has a significant influence on the properties of asphalt binder. This results in a reduction in binder viscosity and an increase in binder stiffness. This, in turn, promotes enhanced aggregate-asphalt binder bonding. Geopolymer refers to a class of inorganic materials characterized by the formation of a long-range, covalently bonded non-crystalline skeleton. The aim of this study was to explore the potential of incorporating waste and by-product materials, specifically fly ash geopolymer additive, in order to influence the microstructure of asphalt binder. The focus was on examining how the inclusion of fly ash geopolymer could alter the internal arrangement and composition of the asphalt binder, leading to potential improvements in its properties and performance. Laboratory experiments were performed to analyze the microstructure of 60/70 and 80/100 asphalt binder samples using Scanning Electron Microscope (SEM) imaging. These samples were modified with various concentrations (3%, 5%, 7%, 9%, and 11%) of fly ash geopolymer additive. The SEM images were obtained to examine the morphological changes and assess the distribution of the fly ash geopolymer particles within the asphalt binder matrix at different additive concentrations. The NOVA NANOSEM 230 equipment was utilized to determine the morphological characteristics of the asphalt binders. The results showed notable variations in the properties of the asphalt binders modified with fly ash geopolymer compared to the unmodified control asphalt binder. The morphological evaluation revealed thorough blending of the fly ash geopolymer additive. Notably, the micrographs demonstrated a denser structure with increased percentages of fly ash geopolymer, indicating a presence of fly ash and alkaline activator promotes the rapid formation of polymerization. These findings emphasize the promising potential of fly ash geopolymer as an additive in asphalt binder. The significant effect it has on viscosity when incorporated into modified asphalt binder makes it a valuable candidate for application in the field.

Keywords: Fly ash geopolymer, asphalt binder additive, Scanning Electron Microscope (SEM)

1. Introduction

At present, the primary material utilized for road surfacing is hot mix asphalt (HMA), which is produced at temperatures ranging from approximately 160°C to 180°C [1]. The elevated temperature plays a vital role in ensuring

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sufficient binder coverage and compaction of the aggregate [2]. The aggregate particles provide stability and load-bearing capacity, while the asphalt binder acts as a binder to hold the aggregate together and provide flexibility to the pavement structure [3]. The performance of HMA pavement is directly influenced by the characteristics and properties of the asphalt binder, as well as external factors like environmental conditions including high temperatures and moisture. Various factors play a crucial role in determining the structural performance of asphalt pavements. To improve the technology associated with HMA, efforts have been made to incorporate additives into the mixture. The purpose of these additives is to enhance the structural integrity of the asphalt pavement and address specific performance issues. By incorporating suitable additives, such as polymers, fibers, or other modifiers, the properties of the HMA can be modified to improve its strength, durability, resistance to cracking and rutting, and overall performance under traffic loads and environmental conditions [4]. These advancements aim to ensure the long-term functionality and sustainability of asphalt pavements. In recent years, the asphalt industry has shown considerable interest in geopolymer, an innovative type of cementitious material, due to its ability to reduce CO2 emissions during the asphalt manufacturing process.

The concept of geopolymer was initially introduced in 1978 [5]. Geopolymers consist of polycondensation molecules with a general structural formula of Mn [-(Si-O2-) z-Al-O-) n. wH20, where M represents sodium (Na) or potassium (K), and n denotes the number of water molecules in the crystalline structure. The basic building blocks of geopolymer, the silicon-oxygen tetrahedron and the aluminum-oxygen tetrahedron, are interconnected through oxygen atoms [6]. Moreover, various industrial waste materials such as coal gangue, fly ash, tailings, and slag can be employed as raw materials for geopolymer synthesis. This innovative utilization of geopolymers as additives in hot mix asphalt (HMA) is expected to offer a viable solution for incorporating industrial waste while enhancing the performance of asphalt mixtures across different binder grades. Implementing this approach is essential for optimizing the utilization of solid waste resources and advancing environmental protection initiatives.

The incorporation of by-product materials like fly ash geopolymer in asphalt influences its properties, resulting in enhanced aggregate-bitumen bonding through reduced binder viscosity and stiffness [7]. Geopolymers, which are inorganic compounds characterized by a long-range, covalently bonded non-crystalline structure, play a crucial role in this process. Utilizing geopolymer in binder modification leads to reduced asphalt viscosity and improved aggregate coating [8]. Consequently, the incorporation of geopolymers in binder modification enhances asphalt performance, inclusion of these additives enhances their sustainability and contributes to mitigating the environmental impact associated with roadways [9]. Other than that, the environmental factors like high temperatures can cause pavement layers to perform poorly, the use of HMA technologies in the manufacture of asphalt pavement needs to be modified to improve pavement performance by selecting the right asphalt binder. The characteristics of the asphalt binder determine how well the hot mixed asphalt pavement performs [10]. Rutting, deformation and moisture damage is known as a major distress in HMA pavements [11]. Use of different geopolymer modified asphalt and optimizing of mixture graduation can help to enhance performance of pavement in HMA technologies. HMA technologies are using high temperature which is the temperature range of 140º C to 180º C. This high temperature is using to ensure the aggregate completely dry and the asphalt binder coating perfectly in asphalt production.

Additionally, Nazar et al. [12] investigated the geopolymerization potential of size-fractionated fly ash. The results showed that distinct fly ash size fractions collected from various hoppers had chemistry, mineralogy, particle size distribution, and glass concentration that varied significantly, leading to different geopolymer properties. The geopolymerization reaction method resulted in the production of a polymer backbone made up of silicon and aluminum atoms [13]. For the geopolymer backbone matrix, the amorphous X-ray equivalent of the tetrahedral alkali aluminosilicate structure has also been proposed [14]. Investigations on Class F fly ash geopolymer's compressive strength and microstructural properties were conducted [15]. As a result, it was discovered that using geopolymer made from Class F fly ash was both environmentally safe and energy efficient.

The objective of this study is to evaluate how different levels of fly ash geopolymer content affect the morphology characteristics of asphalt binders with varying grades. Geopolymers, which are inorganic compounds, form a non-crystalline skeleton with long-range covalent bonding.

2. Materials and Methods

This section describes the materials and method used in this study. To obtain the precise results, the test was conducted strictly followed the standard and specification of ASTM and AASHTO. The asphalt binder 60/70 and 80/100 penetration grades were used in this research are Chevron and Dorotech. Asphalt binder is graded based on range of consistency at standard temperature. The fly ash utilized is class F fly ash, and Table 1 contains a summary of the chemical components of fly ash. The details of the binder used are displayed in Table 2.

Table 1 - Chemical composition of fly ash (%)

Constituent	Fly ash
SiO_2	57.20
Al_2O_3	23.50
Fe_2O_3	3.80
CaO	9.3
MgO	1.0
SO_3	0.20
Na2O	2.43

Table 2 - The specification of the binder

No	Type of test	Method	Specification
1	Softening Point (C)	ASTM D36-2005	-
2	Penetration at 25ºC	ASTM D5-2005	60/70, 80/100
3	Rotational Viscosity (RV)	ASTM D4402-2005	Min 100
4	Ductility	ASTM D113	-

2.1 Preparation of Geopolymer Additive

Geopolymer was formed by combining fly ash and the alkaline medium. The alkali medium used in this study consisted of two components: sodium silicate (Na2SiO3) and sodium hydroxide (NaOH) pellets. These components were separately diluted in distilled water. Sodium silicate was diluted to prepare a sodium silicate solution, while sodium hydroxide pallets were diluted to prepare an 8 Molar (8M) NaOH solution. To activate the alumino-silicate precursors present in fly ash, a mixture of sodium silicate solution and sodium hydroxide solution was prepared. This activation process facilitates the formation and development of the desired geopolymer structure in the fly ash. The mixing procedure is schematically shown in Fig. 1. To create the alkaline medium, a mixture was prepared using sodium hydroxide (8M) and sodium silicate solution in a mass ratio of 100:50%. This ratio ensures the appropriate concentration and composition of the alkaline medium for the desired application. Sodium hydroxide serves as an activator, while sodium silicate contributes to the formation of the geopolymer matrix. The specific concentrations and ratios were chosen based on previous research and considerations for the desired properties and performance of the geopolymer.

Secondly, a mixture of 200 grams of fly ash powder and 80 grams of alkaline medium was blended for a duration of 6 minutes. Subsequently, the resulting slurry was poured into molds with dimensions of 50mm x 50mm x 50mm. After the preparation of the samples, they were left to cure at room temperature (25°C) for a period of 24 hours, followed by additional curing in an oven at 60°C for another 24 hours. This curing process allows the geopolymer binder to develop and solidify, ensuring the desired properties and microstructure formation. Subsequently, the prepared samples were subjected to milling to transform them into powders with smaller particle sizes. This milling process involves grinding or crushing the samples to achieve a fine and uniform powder consistency. The purpose of milling is to enhance the homogeneity of the samples and facilitate further analysis or characterization.

Following the milling step, the powders were sieved to remove any particles with a diameter larger than 0.15mm. This sieving process helps to ensure that the final samples consist of particles within the desired size range, allowing for more accurate analysis and eliminating any potential inconsistencies or outliers caused by larger particles. By sieving the samples, a more standardized and uniform sample set is obtained, enabling reliable and consistent results during subsequent testing and evaluation [16].

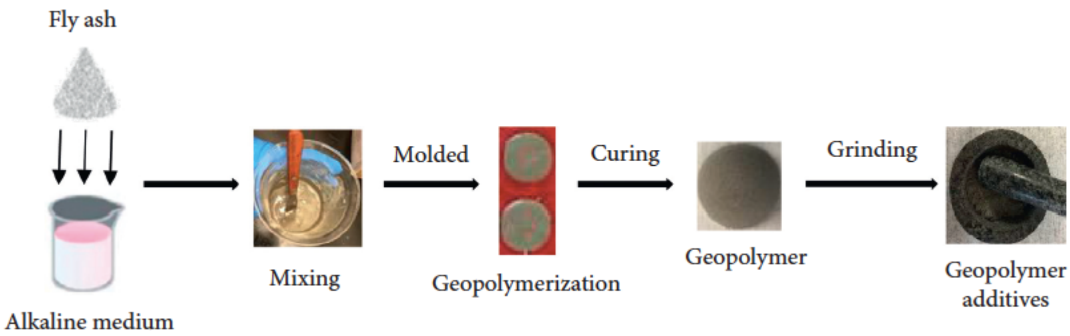

Fig. 1 - Preparation of geopolymer additives [6]

2.2 Materials and Sample Preparation

The specimens in this study were prepared using penetration grade 80/100 and 60/70 binders. Six levels of fly ash-based geopolymer content were tested in the experiments: 0%, 3%, 5%, 7%, 9%, and 11% by mass of the binder. According to ASTM C618 criteria, the fly ash utilized in the study was a class F fly ash and came from the Kapar Power Station in Selangor, Malaysia. The procedure of blending the asphalt binder and fly ash-based geopolymer with a Silverson mechanical mixer is shown in Fig. 2. The base asphalt binder was heated to 110°C in an oven before being combined with the fly ash-based geopolymer. Different additive percentages in the range of 0%, 3%, 5%, 7%, 9%, and 11% by mass of the binder were added to the mixture to guarantee uniformity, and the mixture was then rotated at a speed of 2000 rpm for 120 minutes at a constant temperature of 150°C [17]. The binder was swirled for around 2 minutes prior to adding the addition to ensure a uniform temperature distribution. Table 3 tabulated the blending binder protocols for both asphalt binder grades.

Fig. 2 - Silverson mechanical mixture

Table 3 - Blending binder protocols for both grade asphalt binders

Asphalt Weight (g)	Percent of Asphalt (%)	Fly Ash Weight (g)	Mixing Time (hrs)	Mixing Speed (rpm)	Mixing Temp. (°C)
400	0	0	2	2000	±150
400	3	12	2	2000	±150
400	5	20	2	2000	±150
400	7	28	2	2000	±150
400	9	36	2	2000	±150
400	11	44	2	2000	±150

2.3 Test Method

The scanning electron microscope (SEM) is a sophisticated instrument utilized for the examination and analysis of microstructural morphology and chemical composition. It offers the ability to generate high-resolution images, enabling detailed characterization and understanding of materials at a microscopic level [18]. In this study, the SEM was utilized to acquire a more comprehensive understanding of the morphology of both fly ash and fly ash geopolymer. High-resolution microscopic images generated by the SEM were utilized to illustrate the variations in the reaction area and analyze the microstructure. The surface morphology and microstructure changes in all modified binders were observed and analyzed using the NOVA NANOSEM 230 equipment as shown in Fig 3. The Scanning Electron Microscope (SEM) utilizes signals resulting from interactions between electrons and the sample to visualize and analyze various aspects, including the external morphology (texture), chemical composition, crystalline structure, and material orientation within the samples. This powerful imaging technique provides valuable insights into the microscopic properties of materials [19].

To facilitate sample characterization, it was necessary to prepare the asphalt binder samples and the fly ash geopolymer modifier powder into smaller sizes. For surface analysis, the samples were positioned horizontally on the substrate holder, while for cross-sectional examination, they were placed vertically. A comparison was made between the microstructures of samples containing fly ash geopolymer and those of the control asphalt binder.

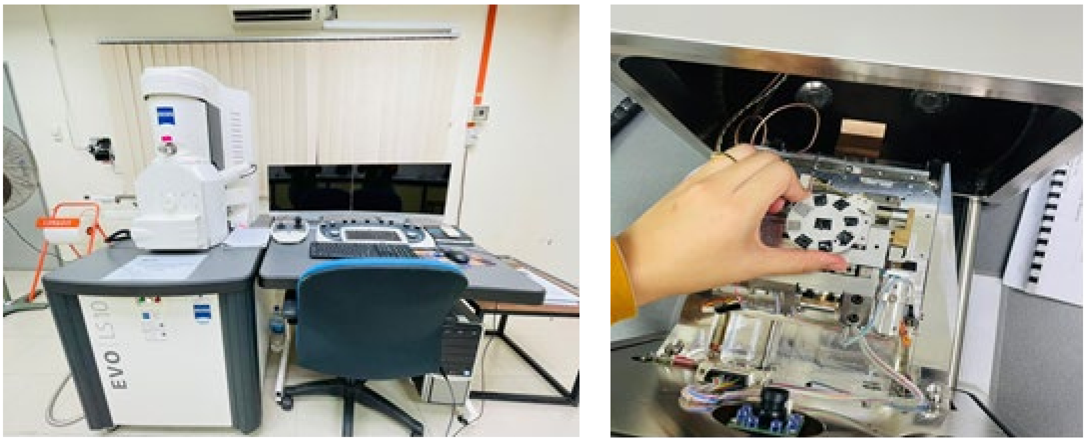

Fig. 3 - NOVA NANOSEM 230 equipment

3. Results and Discussion

The surface morphology, microstructure changes in all modified binders, and physical dispersion of fly ash geopolymer particles were examined and analyzed using scanning electron microscopy (SEM) analysis. The SEM analysis was conducted at a magnification of 2000x for the control fly ash and fly ash geopolymer, and at a magnification of 3000x for the modified binder with different binder grades. Fig. 4 shows the original microstructure of the fly ash used to create the fly ash geopolymer, as observed through SEM analysis. The morphology of the fly ash particles was observed to be predominantly spherical in shape. The original microstructure of the fly ash closely aligned with the description provided by Xu et al. [20], although there were some variations in form, ranging from rounded to angular, particularly in certain mullite and iron crystals within the fly ash particles. Moreover, the surface texture of the fly ash geopolymer microstructure exhibited a range from smooth to dense or highly porous. In certain cases, the surfaces also displayed coatings, such as magnetite. These variations in surface texture indicate different levels of porosity and the presence of additional compounds on the surface of the fly ash geopolymer particles.

Fig. 5 displays the microstructure images of fly ash geopolymer prepared in an alkaline medium. The microstructure analysis reveals the presence of unreacted and partially unreacted particles in proximity within the fly ash geopolymer microstructure. This suggests that the polymerization process of the fly ash geopolymer is not fully complete, and there are areas where the particles have not undergone complete chemical reactions. The polymerization process is significantly influenced by the concentration of calcium present in the fly ash. It has been observed that fly ash with a lower calcium content, such as class F fly ash, is more suitable for the formation of geopolymers compared to fly ash with a higher calcium content, such as class C fly ash. According to Zhao et al. [21], the reaction between fly ash and the alkaline liquid was found to be enhanced when a mixture of sodium hydroxide solution and sodium silicate solution was used. Hence, it can be inferred that sodium silicates and sodium hydroxide actively participated in the polymerization process.

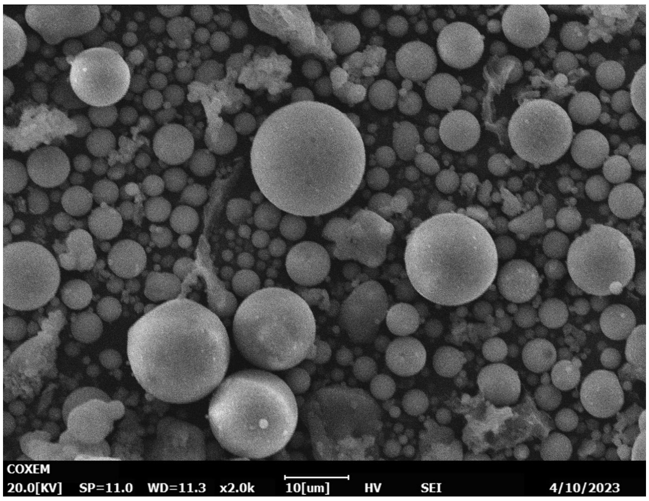

Fig. 4 - SEM image of control fly ash class F

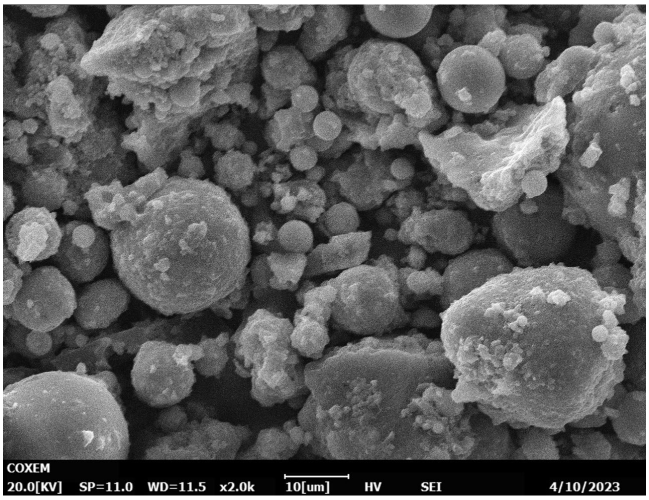

Fig. 5 - SEM image of fly ash geopolymer

Fig. 6 presents the microstructure images of unmodified binders specifically for asphalt binder grade 80/100, while Fig. 7 displays the microstructure images of binders modified with fly ash geopolymer specifically for asphalt binder grade 60/70. The microstructure analysis of asphalt binders containing 3%, 5%, 7%, 9%, and 11% fly ash geopolymer was conducted using SEM. The microstructure images of the asphalt binders modified with fly ash geopolymer exhibit noticeable differences compared to the microstructure of the control asphalt binder. These differences indicate the influence of the fly ash geopolymer on the microstructural characteristics of the modified binders. The microstructure images reveal that at higher percentages of fly ash geopolymer, the microstructure of the base binder appears larger in size compared to the fly ash modified asphalt binder. This indicates that the addition of fly ash geopolymer leads to a finer and more compact microstructure in the modified asphalt binder. Morphological evaluation confirms thorough blending of the fly ash geopolymer additive. Significantly, the micrographs demonstrate a denser formation with an increased percentage of fly ash geopolymer, indicating a rapid polymerization process facilitated by the presence of fly ash and the alkaline activator. The results indicate that fly ash geopolymer has significant potential for application in asphalt binder. The incorporation of fly ash geopolymer leads to notable effects on the viscosity of the modified asphalt binder, resulting in a more compact microstructure. This suggests that fly ash geopolymer can contribute to improved performance and properties of the asphalt binder.

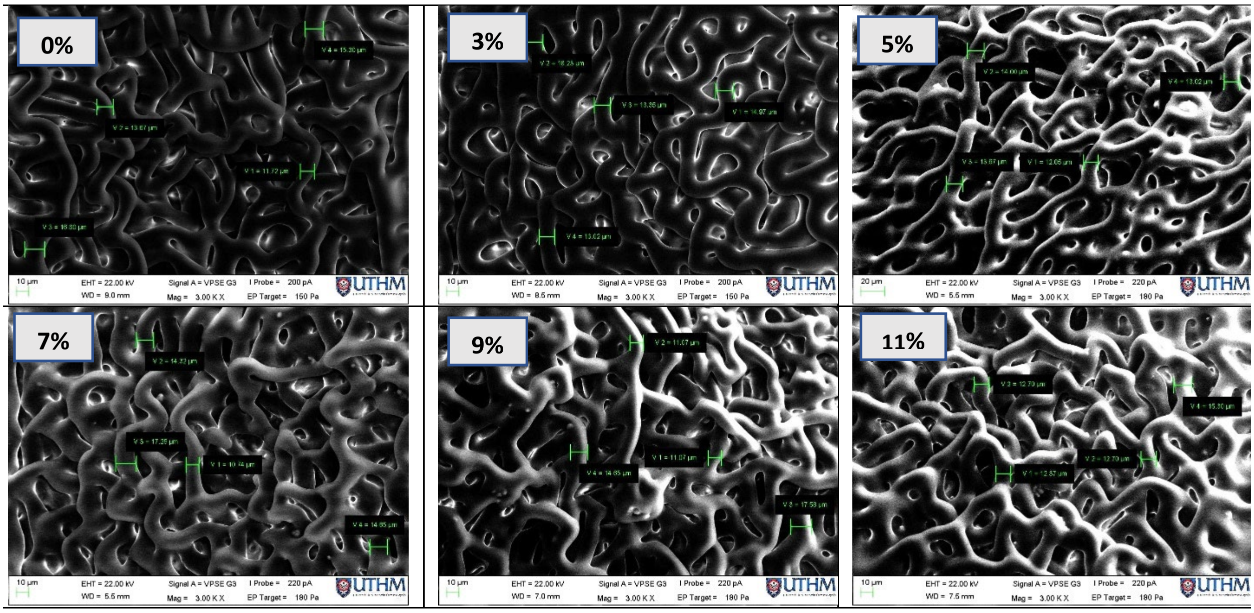

Fig. 6 - SEM image of asphalt binder grade 80/100 at 3000 magnifications

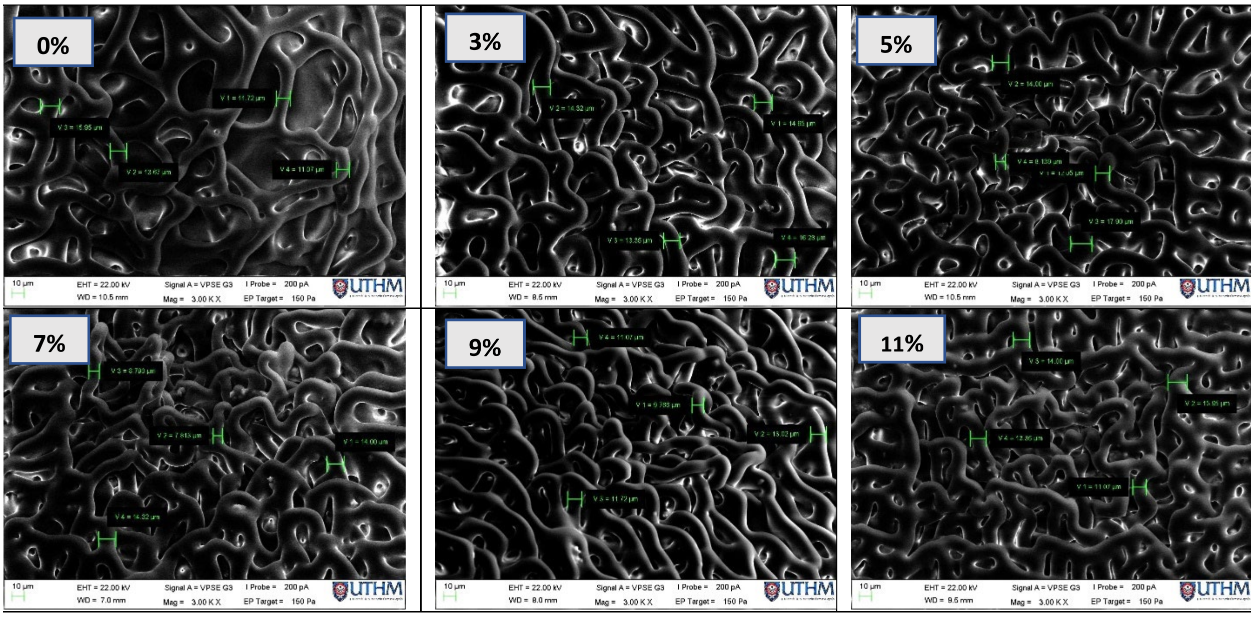

Fig. 7 - SEM image of asphalt binder grade 60/70 at 3000 magnifications

4. Conclusions

The SEM analysis shows that the addition of fly ash geopolymer to the asphalt binder has resulted in notable changes in the microstructure compared to the control binder. The SEM micrographs indicate that the presence of calcium content in the materials significantly influences the formation of calcium silicates and geopolymer gel products. In this context, it can be inferred that sodium silicates and sodium hydroxide actively contribute to the polymerization process. Their presence indicates their complete participation in the polymerization reactions, leading to the observed changes in the microstructure of the fly ash geopolymer modified asphalt binder. The SEM analysis also revealed that the fly ash geopolymer additive improved the adhesion between the aggregate and the asphalt binder. This is attributed to the formation of a strong bond between the geopolymer and the aggregate particles, which helps to prevent the loss of aggregate and ultimately enhances the durability of the asphalt pavement. In addition, the use of fly ash geopolymer additive can help to reduce the amount of asphalt binder required for road construction, which can result in cost savings and a reduction in greenhouse gas emissions associated with asphalt production [22]. Therefore, the use of fly ash geopolymer additive in asphalt binder has the potential to enhance the performance and sustainability of asphalt pavement while helps to reduce their environmental impact.

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References

[1] Omranian S. R., Hamzah M. O., Mounik G. S., Reddy K. P., Kapadia Y. M. & Teh S. Y. (2020). Laboratory investigation on warm mix asphalt incorporating organo-silane additive. IOP Conference Series Earth and Environmental Science, 463, 012068.

[2] Arshad A. K., Shaffie E., Ismail F., Hashim W. & Mustapa N. S. F. (2020). Warm mix asphalt surfacing performance for different aggregate gradations with Cecabase RT additive. IOP Conference Series Earth and Environmental Science, 498, 012019.

[3] Al-akhaly I. A. (2018). Engineering properties of basalt coarse aggregates in Hamdan area, NW Sana' a, Yemen. Journal of Geological Engineering, 42(4), 159-174.

[4] Mohammad Taher M. N., Aman M. Y., Abdullahmusa N. F. A. (2018). Physical properties and chemical bonding of advera ® modified asphalt binder. MATEC Web of Conferences, 250, 02008.

[5] Tang N., Deng Z., Dai J. G., Yang K., Chen C. & Wang Q. (2018). Geopolymer as an additive of warm mix asphalt: Preparation and properties. Journal of Cleaner Production, 192, 906-915.

[6] Davidovits J. (2011). Geopolymer chemistry and applications. Geopolymer Institute.

[7] Hamid A., Alfaidi H., Baaj H. & El-hakim M. (2020). Evaluating fly ash-based geopolymers as a modifier for asphalt binders. Advances in Materials Science and Engineering, https://doi.org/10.1155/2020/2398693.
[8] Yin Y., Chen H., Kuang D., Song L. & Wang Lu. (2017). Effect of chemical composition of aggregate on interfacial adhesion property between aggregate and asphalt. Construction and Building Materials, 146, 231-237.
[9] Hoy M., Horpibulsuk S., Rachan R., Chinkulkijniwat A. & Arulrajah A. (2016). Recycled asphalt pavement fly ash geopolymers as a sustainable pavement base material: Strength and toxic leaching investigations. Science of the Total Environment, 573, 19-26.
[10] Junaid M., Irfan M., Ahmed S. & Ali Y. (2018). Effect of binder grade on performance parameters of asphaltic concrete paving mixtures. International Journal of Pavement Research and Technology, 11(5), 435-444.
[11] Li N., Zhan H., Yu X., Tang W., Yu H. & Dong F. (2021). Research on the high temperature performance of asphalt pavement based on field cores with different rutting development levels. Materials and Structures, 54, 1-12.
[12] Nazar S., Yang J., Amin M. N., Khan K., Ashraf M., Aslam F. & Eldin S. M. (2023). Machine learning interpretable-prediction models to evaluate the slump and strength of fly ash-based geopolymer. Journal of Materials Research and Technology, 24, 100-124.
[13] Ye Z., Ren W., Yang H., Miao Y., Sun F. & Wang L. (2021). An improved asphalt penetration test method. Materials, 14(1), 147.
[14] Gunasekara C., Law D. W., Setunge S., Burgar I. & Brkljaca R. (2017). Effect of element distribution on strength in fly ash geopolymers. ACI Materials Journal, 114(5), 795.
[15] Jiang X., Zhang Y., Xiao R., Polaczyk P., Zhang M., Hu W., Bai Y. & Huang B. (2020). A comparative study on geopolymers synthesized by different classes of fly. Journal of Cleaner Production, 270, 122500.
[16] Bujang H., Aman M, Y., Arumugam T. N. & Taher M. N. M. (2023). Physical characterization of modified asphalt binder with differing fly ash geopolymer contents. International Journal of Integrated Engineering, 15(1), 331-338.
[17] Guo X., Shi H. & Dick W. A. (2010). Compressive strength and microstructural characteristics of class C fly ash geopolymer. Cement and Concrete Composites, 32(2), 142-147.
[18] Karyaman D. M. A., Ekaputri J. J., Widyatmoko S. & Ahyudanar E. (2020). The effect of various NA2SiO3/NAOH ratios on the physical properties and microstructure of artificial aggregate. Journal of Engineering Science and Technology, 15(2), 1139-1154.
[19] Dayal S. & Soundarapandian N. (2018). Effect of fly-ash based geopolymer coated aggregate on bituminous mixtures. Journal of the Croatian Association of Civil Engineers, 70(3), 187-199.
[20] Xu J., Yang E., Luo H. & Ding H. (2020). Effects of warm mix additives on the thermal stress and ductile resistance of asphalt binders. Construction and Building Materials, 238, 117746.
[21] Zhao Z., Jiang J., Ni F., Dong Q., Ding J. & Ma X. (2020). Factors affecting the rutting resistance of asphalt pavement based on the field cores using multi-sequenced repeated loading test. Construction and Building Materials, 253, 118902.
[22] Bujang H., Aman M. Y. & Taher M. N. M. (2022). Volumetric properties of asphalt mixture containing fly ash geopolymer. Conference Series Earth and Environmental Science, 1022(1), 012034.