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Statistical Approach of Additive Effect on Volumetric Characteristics of Asphalt Mixtures †

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Abstract: The use of additives in asphalt concrete has improved the performance of road pavements. Some additives include silica powder, fly ash, and crumb rubber. This study analyzed additive levels' effect on the volumetric characteristics of asphalt mixtures using a statistical approach. This research uses aggregate gradation for asphalt concrete wearing a coarse layer to determine the effect of additive content on the volumetric of hot asphalt mixtures. Asphalt modification is used with silica powder at compositions of 0%, 2%, and 6%; fly ash with 0%, 2%, and 4%; and crumb rubber with 0%, 1%, and 1.25%. Samples were made and tested according to the Marshall test procedure. The volumetric characteristics of the asphalt mixture were subjected to correlation analysis with the Pearson and Spearman rank approaches and the ANOVA test. The Pearson and Spearman rank correlation analyses showed that fly ash and silica additives were suspected of having no correlation and influence on volumetric characteristics except for crumb rubber. The ANOVA test results show that the largest volumetric characteristics are significantly influenced by the crumb rubber additive on voids in mineral aggregates. In contrast, the silica and fly ash additives affect voids in mineral aggregates and Marshall stability. The difference in silica and fly ash additive content has shown different ANOVA test results even though they have the same size and composition of the forming elements.

Keywords: additive; volumetric; correlation; ANOVA

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1. Introduction

In road pavement design, it is essential to fulfill the specified technical requirements so that the road pavement structure can accept traffic loads during the design life. Applying these technical requirements ensures that each material mixture achieves a specific performance value according to design needs. Material limitations and environmental conservation efforts have required researchers to utilize waste as additional materials. Many previous studies have discussed the composition of material mixtures and their additives to obtain material properties that meet the specified technical specifications. Adding additives to asphalt mixtures can improve pavement performance, such as durability against weather from peeling or against traffic loads that work [1].

The development of material technology has used nano-sized materials. The process of forming materials to very small sizes requires certain technology. The use of nanomaterials has produced mixtures that have higher resistance to damage [2]. Several researchers have used nano-sized materials to improve the performance of hot asphalt mixtures for road pavements. One study using nano-sized silica powder additives has been shown to increase stiffness, increase elastic properties, and reduce the susceptibility of binder temperatures [3]. In other studies, the size of additives derived from mineral materials is the object of a more detailed review. The combination of micro- and nano-sized additives in lime has shown an increase of 60% in the ratio of hot mix asphalt aggregate tensile strength [4]. Another additive derived from minerals that is often studied is fly ash. The use of this material has been proven not only to improve the performance of the mixture added but also to overcome waste from coal combustion. The role of fly ash as an additive has increased the complex modulus (G*), which indicates hardening in the asphalt mixture [5]. The use of fly ash has shown an increase in the stability properties of hot mix asphalt concrete and has shown higher performance compared to fillers for 5–8% content [6].

Introducing an innovative approach, we explore using crumb rubber derived from car tire waste. This material, crushed to nano size, serves as an additive in asphalt concrete mixtures, presenting a novel solution to the challenge of waste management in the construction industry.

Nano-crumb rubber (NCR) has changed the performance of pure asphalt mixtures by increasing the deformation rate and dynamic stability [7]. Utilizing car tire waste requires a particular process so that this material can be used as a micro- or nano-sized additive. The crumb rubber additive has changed the softening point of the asphalt to be higher so that it is more resistant to the effects of environmental temperature [8]. The size of the crumb rubber has shown different performance in asphalt concrete mixtures. A crumb rubber size of 0.15 mm has shown an increase in the indirect tensile strength value even though it reduces the humidity stage [9].

The mechanism for mixing additives into asphalt mixtures consists of two types, namely, the dry process and the wet process. The dry process involves mixing additives into aggregates before mixing them with asphalt. In contrast, the wet process involves mixing additives into asphalt before mixing them with aggregates [10]. Both methods are selected according to the characteristics of the additives. Generally, fiber-shaped additive materials are easier for fine-grained materials to use in the dry and wet processes. The additives used in the dry process function to reduce the volume of asphalt as a binder attached to the aggregate [11]. In the wet process, several control parameters are required, such as mixing temperature, mixing rate, and mixing time, which will cause the rheological properties of the asphalt to change [12]. Additives of smaller sizes will easily mix with asphalt through the wet process. Additives with high melting points are not easily mixed homogeneously with asphalt, making them more suitable for the dry process [10].

This study of the Marshall test results, a volumetric parameter characteristic of asphalt aggregate mixtures, provides practical insights. Drawing from various previous studies, the impact of additives on asphalt mixtures with dry processes, particularly the widely studied plastic waste material as an additive in cement concrete and asphalt concrete mixtures.

The use of high-density polyethylene (HDPE) has reduced the density and voids filled with asphalt (VFA) values by 3–7% each, increased the void in mixture (VIM) value by 9%, and the void in mineral aggregate (VMA) value by 2%. Marshall test data show that adding low-density polyethylene (LDPE) plastic to the asphalt mixture asphalt concrete wearing course (AC-WC) penetration 60/70 can affect the increase and decrease in VIM, VMA, and VFA values [13–15]. Using nano-sized additives in asphalt mixtures with a wet process also affects the volumetric characteristics.

Asphalt mixtures with nanocarbon black additives reduce the VIM value by 47% due to increased cohesion in the asphalt and reduce the VMA value by about 12% due to adhesion between the asphalt and aggregate [16]. In addition to affecting the volumetric characteristics, nano-sized additives, through a wet process, change the physical properties of asphalt. One example is using 5% nano-silica, which increases the viscosity value by 60% at a temperature of 135° [17]. The viscosity value is essential for asphalt functions as a determinant of the temperature of the mixing and compaction process of the asphalt concrete mixture. The increase in the physical and mechanical properties of asphalt mixtures is directly proportional to the increase in the content of polymer additives [18]. The effect of crumb rubber additives on the AC-WC mixture impacts the decrease in VMA, VIM, VFA, Marshall stability (MS), and Marshall quotient (MQ) values [19,20]. The difference in characteristics is caused by the content of tire rubber waste, which affects various properties of the asphalt mixture.

Several statistical methods can determine the characteristics of asphalt concrete mixtures. Advanced statistical analysis can be used for laboratory data processing. Advanced statistical analysis methods include Pearson correlation and Spearman's rank correlation coefficient [21]. The analysis of variance (ANOVA) test results revealed that the variable pair's influence was statistically significant [22]. The F value can be used to compare the effects of additive materials on the moisture resistance of asphalt concrete mixtures. The ANOVA test can also be used to examine the aging process of rolling thin-film oven (RTFO) asphalt through the DSR test. Research used the output parameters of the ANOVA to determine that the relationship between the influence of one pair of variables is greater than the other [23–25]. The analysis process in the ANOVA test can be divided based on factors, including the asphalt mixture's volumetric characteristics and mechanical properties [26]. These analyses determine the relationship between two variables and the magnitude of the influence of the relationship between the pair of variables and other pairs of variables. This study analyzes the effect of additive levels derived from minerals and fibers on the volumetric characteristics of the asphalt mixture. Data were analyzed using a statistical approach: correlation analysis and ANOVA test. Thus, this study contributes to the knowledge of using mineral or fiber additives in asphalt mixtures.

2. Material and Method

This study used additional materials for asphalt concrete mixtures: silica, fly ash, and crumb rubber. The mixture's aggregates are from the Asemindo Group located in Purwakarta, Indonesia. The asphalt used comes from Pertamina's production, located in Cilacap, Indonesia. Additional materials are in the size of micro-silica (MS), micro-fly ash (MFA), and micro-crumb rubber (MCR) sieve #200 and through the milling process.

The research stages can be seen in Figure 1. Stage 1 is to prepare materials in the form of additives (1a), asphalt (1b), and aggregate (1c). Stage 2 is to make modified asphalt with the three additives. Stage 3 is to make Marshall test objects in five asphalt contents with pure asphalt and three types of modified asphalt. Stage 4 tests the volumetric characteristics of asphalt mixtures at the University of Indonesia Structure and Materials Laboratory. Stage 5 is the analysis of the volumetric characteristics of asphalt mixtures against the use of additive content using a statistical approach.

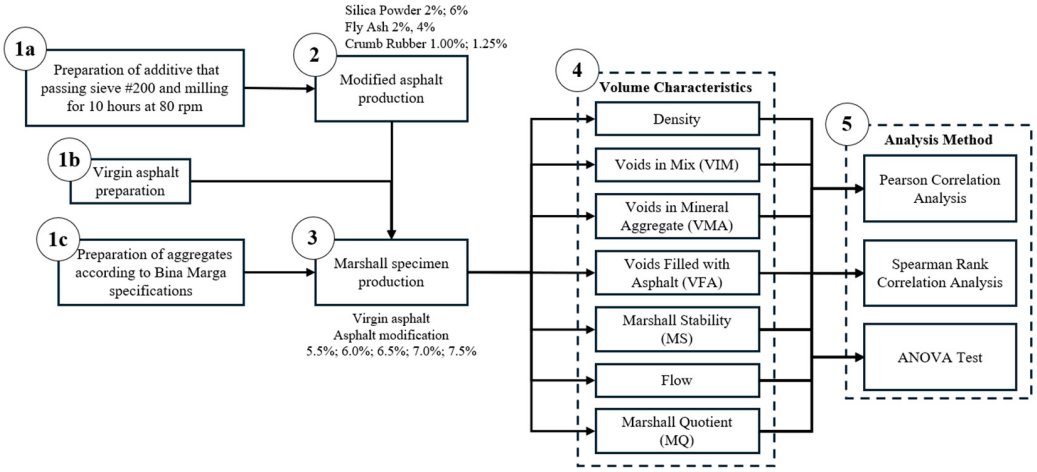


Figure 1. Research flow chart.

2.1. Aggregate

Figure 2 shows the aggregate, which consists of coarse, medium, and fine aggregates. According to Bina Marga, the aggregate gradation is designed to be between the upper and lower limits of the AC-WC specifications, as shown in Figure 3.



Figure 2. Aggregate after oven: (**a**) coarse aggregate, (**b**) medium aggregate, and (**c**) fine aggregate.

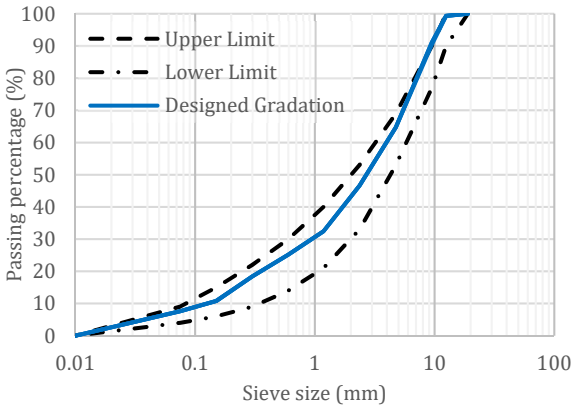


Figure 3. Aggregate distribution.

2.2. Material Additives

The additional materials used in this study consisted of three types: silica powder, fly ash, and crumb rubber. The three additional materials have been filtered #200 and milled to a size of <100 µm. As shown in Figure 4, the Mini Ball Mill tool is assisted by five iron balls with a diameter of 15 mm (weighing 42.04 g) and five iron balls with a diameter of 8 mm (weight = 20.88 g). This milling was performed for 10 h with a maximum rotation speed of 80 rpm.

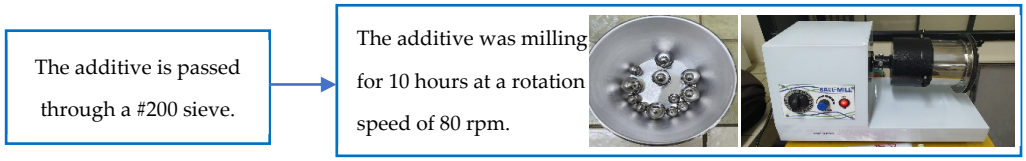


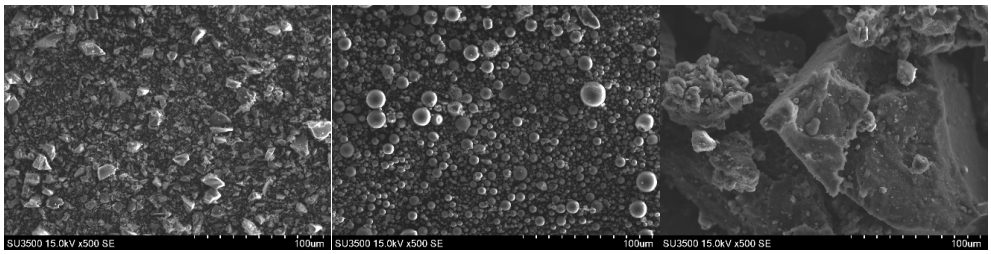
Figure 4. Mini ball mill.

The content of silica powder and fly ash is dominated by O_2 and SiO_2 while $CaCO_3$ dominates crumb rubber. The composition of the additive-forming elements can be seen in Table 1.

Table 1. Additive chemical content.

Element	Weight (%)		
	Silica Powder	Fly Ash	Crumb Rubber
O_2	90.7	53.97	-
$CaCO_3$	-	-	78.70
SiO_2	34.41	18.86	13.74
Al_2O_3	2.68	9.45	0.34
Fe	2.07	4.89	0.22
Wollastonite	1.60	6.55	1.77
Ti	1.53	-	-
MAD-10 Feldspar	0.76	1.02	0.13
Albite	0.51	2.45	-
MgO	0.15	2.83	0.09

Measurement of additive dimensions through SEM-EDS test procedures with 500× magnification using Hitachi SU3500 at the Advanced Characterization Laboratory, National Research and Innovation Agency. The Hitachi SU3500 is manufactured by Hitachi High-Technologies Corporation, headquartered in Tokyo, Japan. The morphological properties of fly ash are different from those of silica powder, which is sharp–angular, while the morphology of crumb rubber appears to be lumpy. The morphological appearance of each additive can be seen in Figure 5.



(**a**) (**b**) (**c**)

Figure 5. SEM imaging result of additives: (**a**) silica powder, (**b**) fly ash, and (**c**) crumb rubber.

2.3. Asphalt

The virgin asphalt used is classified as asphalt with Pen 60/70, which is tested at a temperature of 25 °C according to ASTM D-5 [Ref]. The asphalt softening point test results refer to ASTM D-36, showing a value of 48.35 °C. Furthermore, the asphalt flash point test results are based on ASTM D-92, with a result of 281.40 °C. The specific gravity value at a measurement temperature of 25 °C according to ASTM D-70 is 1.070 gr/cm^3.

The process of modifying asphalt is thorough and meticulous. It involves several types of modifications, including (a) virgin asphalt mixed with 2% and 6% silica powder; (b) virgin asphalt mixed with 2% and 4% fly ash; and (c) virgin asphalt mixed with 1% and 1.25% crumb rubber. The process of mixing pure asphalt with additives is a wet process

(Figure 6). Virgin asphalt is heated to a temperature of 140 °C. The additives are then mixed into the asphalt with great care, assisted by Mixer OS-10L with a rotational speed of 2000 rpm. The mixing process is carried out for a full 60 min at a constant temperature, namely at 140 ± 5 °C. The Mixer OS-10L is manufactured by Joanlab, headquartered in Huzhou City, China.

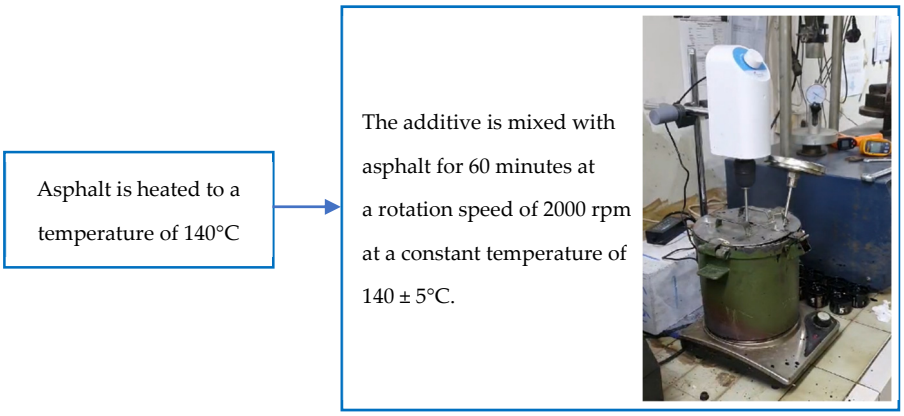


Figure 6. Modified asphalt mixing process.

3. Result and Discussion

3.1. Volume Characteristics of Asphalt Mixture

The volumetric characteristics in this study include VIM, VMA, VFA, and MS. The volumetric characteristics for asphalt mixtures with silica powder compositions containing 0%, 2%, and 6% can be seen in Figure 7. The volumetric characteristics for asphalt mixtures with fly ash compositions of 0%, 2%, and 4% can be seen in Figure 8. The volumetric characteristics for asphalt mixtures with crumb rubber compositions of 0%, 1%, and 1.25% can be seen in Figure 9.

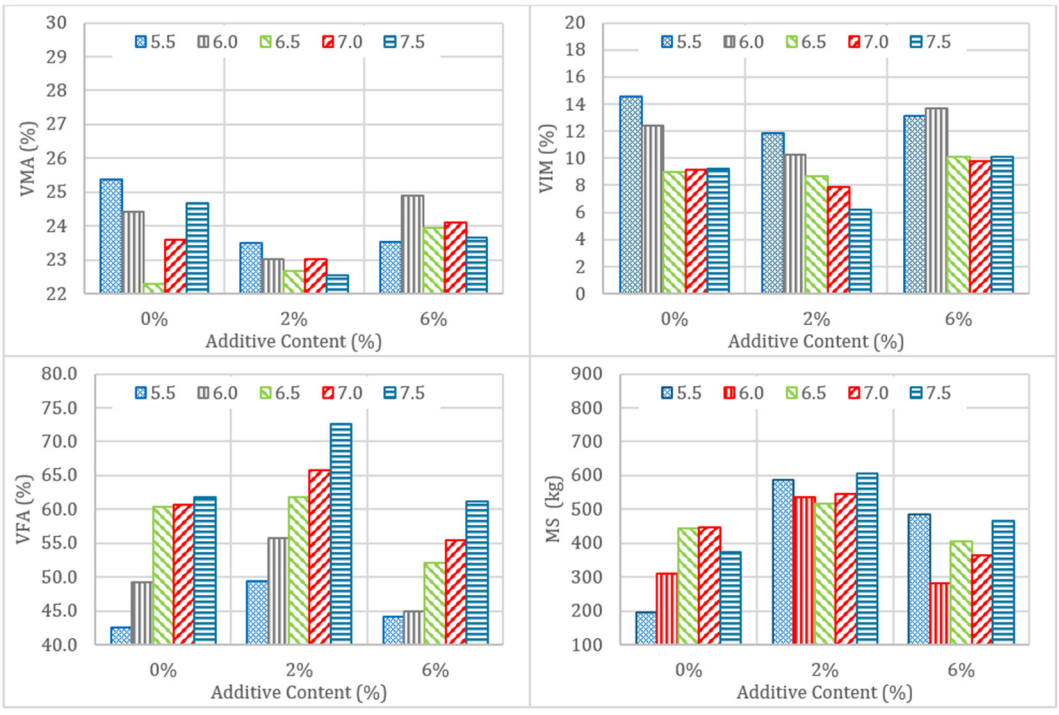


Figure 7. Volumetric characteristics of asphalt mixtures with silica powder additives.

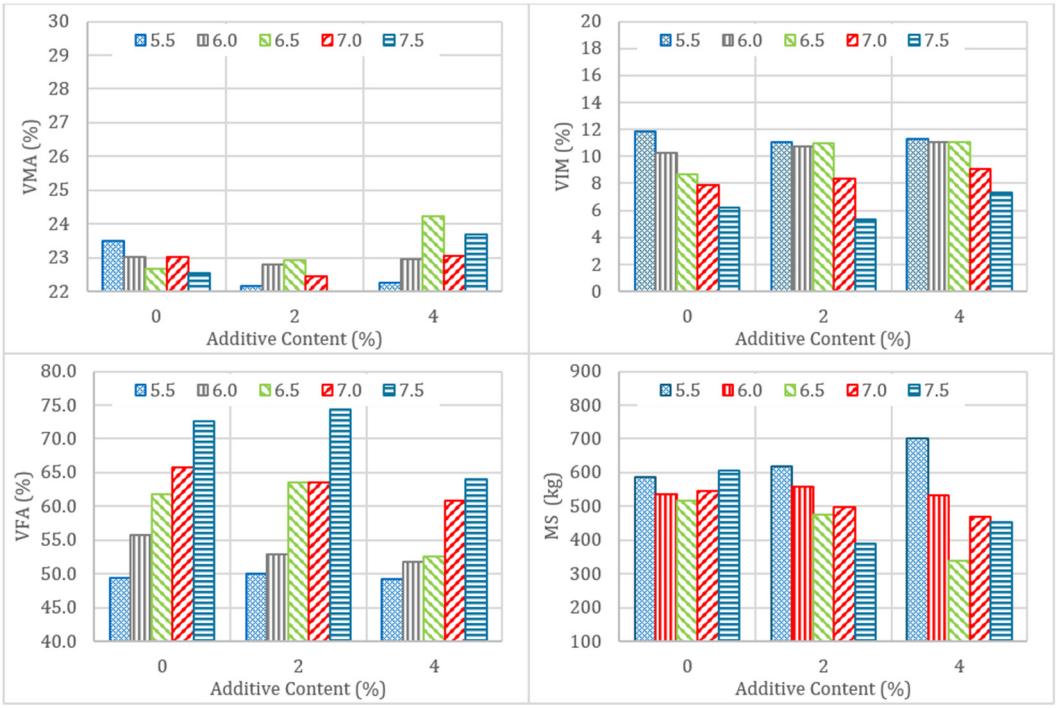


Figure 8. Volumetric characteristics of asphalt mixtures with fly ash additive.

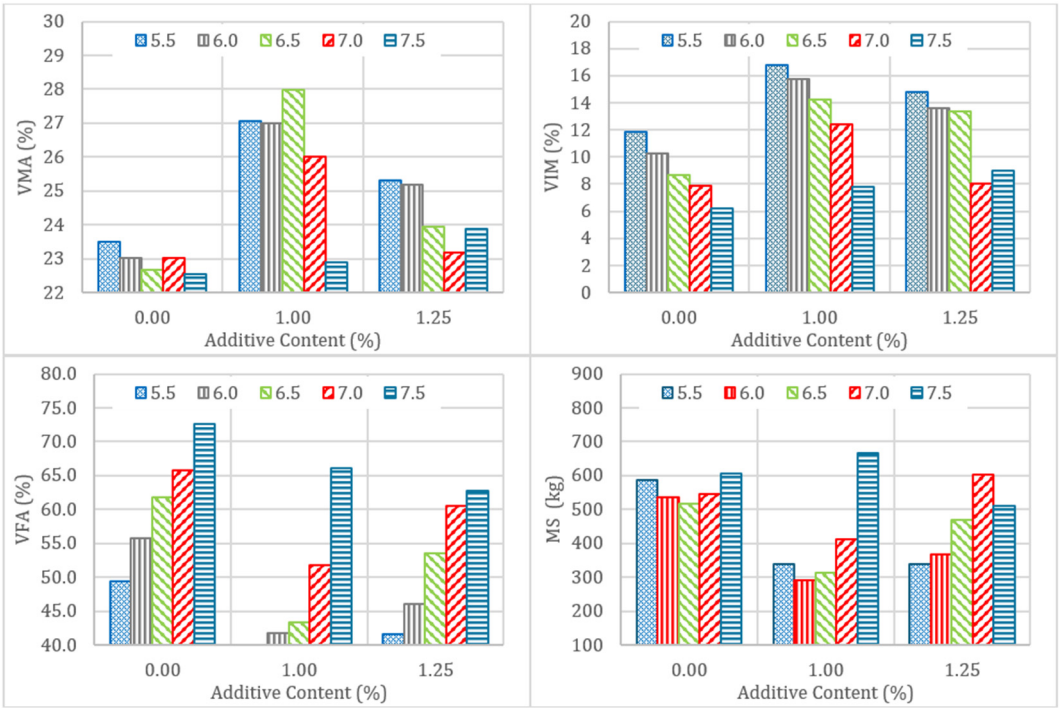


Figure 9. Volumetric characteristics of asphalt mixtures with crumb rubber additives.

The average value of VMA and VIM for each variation of additive content is similar in asphalt mixtures with mineral additives (silica powder and fly ash). In contrast, asphalt mixtures with fiber additives (crumb rubber) appear to form a pattern that can be used to calculate the optimal value of additive content. Volumetric characteristics for asphalt mixtures with mineral additives (Figures 7 and 8) show that the average VFA trend decreases with increasing additive content. In contrast, asphalt mixtures with fiber additives (Figures 7 and 9) show that the average VFA trend tends to increase. Trend MS maximum

average in asphalt mixtures found in mineral additives. Conversely, the minimum value is obtained for fiber additives.

3.2. Correlation Analysis

The correlation value between additive levels and the volumetric characteristics of asphalt mixtures can be seen in Table 2. In contrast, the correlation between the volumetric characteristics of asphalt mixtures and MS values can be seen in Table 3. This correlation value indicates the strength or weakness of the relationship between two variables. The correlation value ranges from −1 to 1. A correlation value > |0.7| means that the correlation between the two variables is strong; a correlation value < |0.4| implies that the correlation between the two variables is weak, and a correlation value between |0.4–0.7| means that the correlation between the two variables is moderate [21]. The Pearson method evaluates the linear relationship of two variables, while the Spearman rank method shows a non-linear relationship [27,28].

Table 2. The correlation value of additive content with volumetric characteristics of asphalt mix.

Additive	Method	Correlation Value						
		Density	VMA	VFA	VIM	MS	Flow	MQ
Silica powder and fly ash	Pearson	0.11	−0.25	−0.03	0	−0.07	−0.18	0.03
	Rank spearman	0.12	−0.25	−0.05	0.07	−0.15	−0.21	−0.01
Crumb rubber	Pearson	0.98 [a]	0.96 [a]	0.87 [a]	−0.96 [a]	0.90 [a]	−0.11	−0.96 [a]
	Rank spearman	0.73 [a]	0.57 [a]	0.78 [a]	−0.78 [a]	0.81 [a]	−0.08	−0.57 [a]

[a] indicates moderate or strong correlation.

Table 3. The correlation between volumetric characteristics of asphalt mixtures with MS.

Additive	Method	Correlation Value					
		MS—Density	MS—VMA	MS—VFA	MS—VIM	MS—Flow	MS—MQ
Silica powder and fly ash	Pearson	0.37	−0.42 [a]	−0.13	0.08	0.42	0.87 [a]
	Rank spearman	0.41 [a]	−0.49 [a]	−0.23	0.21	0.19	0.91 [a]
Crumb rubber	Pearson	0.88 [a]	0.84 [a]	0.98 [a]	−0.92 [a]	0.06	−0.83 [a]
	Rank spearman	0.46 [a]	0.43 [a]	0.96 [a]	−0.98 [a]	0.16	−0.40

[a] indicates moderate or strong correlation.

Table 2 summarizes the correlation values (r) based on each asphalt mixture's Pearson and Spearman rank methods. The results show that no volumetric characteristics correlate with the additive content in asphalt mixtures with silica powder and fly ash. In asphalt mixtures with crumb rubber, there is a correlation between the additive content and its volumetric characteristics, including density ($r_{pearson}$ = 0.98), VMA ($r_{pearson}$ = 0.96), VFA ($r_{pearson}$ = 0.87), VIM ($r_{pearson}$ = −0.96), MS ($r_{pearson}$ = −0.11), and MQ ($r_{pearson}$ = −0.96) correlated with the additive content. Table 3 shows that the volumetric characteristics that correlate with the MS value for asphalt mixtures mixed with additives sourced from silica powder minerals or fly ash are VMA ($_{rrank\ spearman}$ = −0.49) and MQ ($r_{rank\ spearman}$ = 0.91). Asphalt mixture with crumb rubber additive shows that the MS value correlates with all volumetric characteristics, including density ($r_{pearson}$ = 0.88); VMA ($r_{pearson}$ = 0.84); VFA ($r_{pearson}$ = 0.98); VIM ($_{rrank\ spearman}$ = −0.98); and MQ ($r_{pearson}$ = −0.83).

3.3. ANOVA Test

ANOVA Test: A single factor is conducted for several variables, including VIM, VFA, VMA, and MS. This serves to strengthen the results of the correlation analysis regarding the relationship between the variables studied. The F value can be used to compare the influence of one pair of variables with another pair of variables [23]. The greater the F value

on a particular variable, the stronger the additive level influences the variable. The variables that influence this ANOVA test are variables that have optimal values for variations in additive levels.

Table 4 summarizes the F, p, and F_{Crit} values from the ANOVA test results derived from the additive content data group. The results show that the silica powder content in the modified asphalt mixture affects two variables, including VMA and MS. The three variables have a p-value < 0.05 and an F value > F_{Crit} value, so the average values of the two data points from these two variables are statistically different. These data show that the silica powder content influences the values of the two variables. The modified asphalt mixture with fly ash does not affect all volumetric characteristics because all variables have a p-value > 0.05 and the F value < F_{Crit} value, so statistically, it is stated that the average values of the two data points from these two variables are the same. In the previous analysis, the asphalt mixture with silica powder and fly ash additives had the same pattern. In the ANOVA test, the two mixtures could not be classified into one unit. The difference in the influence of additive levels on volumetric characteristics can be influenced by different forms of additive particles (Figure 3) and variations in additive levels. In addition to VMA, the MS variable is considered to be influenced by the silica powder content. However, this variable is weakly correlated with the silica powder content, indicating that the relationship between the two variables with the silica powder content is not directly correlated. Therefore, the ANOVA test identifies MS as a derived variable from a variable that is directly correlated with the silica powder content. Although the silica powder content is weakly correlated with MS. However, changes in the silica powder content as an additive in the wet process have a statistically significant effect. Table 4 states that the F_{VMA} value < F_{MS} value with the conclusion that the silica powder content most strongly influences the VMA value. Another thing that happens in asphalt mixtures with crumb rubber affects VMA.

Table 4. Summary of F and p-values for data groups based on additive levels.

Volumetric Characteristics	n	Asphalt Mixture with Silica Powder		Asphalt Mixture with Fly Ash		Asphalt Mixture with Crumb Rubber		F_{Crit}
		F-Value	p-Value	F-Value	p-Value	F-Value	p-Value	
Density	3	2.69	0.11	0.85	0.45	1.77	0.21	3.89
VIM	3	0.93	0.42	0.15	0.87	2.84	0.1	3.89
VFA	3	0.88	0.44	0.82	0.76	2.22	0.15	3.89
VMA	3	12.06 [a]	0.00 [b]	2.86	0.1	8.27 [a]	0.01 [b]	3.89
MS	3	6.21 [a]	0.01 [b]	3.3	0.07	0.21	0.82	3.89
Flow	3	4.87 [a]	0.03 [b]	0.57	0.58	2.5	0.12	3.89
MQ	3	2.69	0.11	0.85	0.45	3.29	0.07	3.89

[a] indicates value F > F_{Crit}; [b] value $p \leq 0.05$.

4. Conclusions

This study used a modified asphalt mixture with micro-size additives. The results of the study using a statistical approach showed that there was an influence and correlation between the additive content and several volumetric characteristics of the asphalt mixture. The Pearson and Spearman rank correlation analysis results show that the use of silica and fly ash additives does not correlate to all volumetric characteristics except for the use of crumb rubber additives. The results of the ANOVA test show that the crumb rubber additive significantly influences VMA's most considerable volumetric characteristics, while the silica powder and fly ash additives affect VMA and MS. Despite having the same size and composition of constituent elements, the unique characteristics of silica powder and fly

ash additives have shown different ANOVA test results, adding an intriguing dimension to our research.

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