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Pyrolysis of Reclaimed Asphalt Aggregates in Mortar

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Abstract. Asphalt pavement consists of aggregates resulting in a waste material at end of its life. The aggregates can be reused as basic material for asphalt or cementitious binding agents. In both scenarios, the recycled aggregates should provide a good bond with the binder to achieve strength. This study focuses on reusing recycled asphalt aggregates (RAA) in mortar. The major weakness of RAA is the thin oily film originating from the asphalt residue, weakening the bond with cement. The pyrolysis method is accessed in an attempt to overcome this weakness. Three scenarios were investigated; the use of virgin aggregates (VA), RAA, and pyrolysis recycled asphalt aggregate (PRAA) as constituent in mortar. All variables were set a constant except for the aggregate type, the VA mortar function as controlling element. This research is methodologically based on experimental data conducted in the laboratory, while aggregate samples were taken from the field. To analyze the influence of pyrolysis to the aggregate-to-cement bond behaviour, qualitative and quantitative data were collected. The quantitative data were the mechanical properties, the mortar tensile, and compression strength. The qualitative data were obtained from scanning electron microscope readings to visually observe the aggregate surface roughness and voids, including the aggregates cross-section and pre-existing micro-cracks in the aggregate-to-cement interface. Supporting data were the aggregates‘ abrasion rate and absorption. The RAA resulted in a significant mortar strength decrease. This conclusion was supported by the findings of pre-existing cracks in the interfacial transition zone. The pyrolysis method improved the compression strength but negligibly affected the tensile behavior. The compression and tensile strength increased as a function of time for both RAA and PRAA, and a strength convergence was reached at 28 days. The PRAA is considered an option for reuse in mortar, supporting nature conservation.

Keywords: Mechanical properties; Mortar constituent; Pyrolysis recycled asphalt aggregates

1. Introduction

The aggregates in cementitious composites originate from quarries or through stone crushing, which is basically recyclable (Ashadi et al., 2015; Turu’allo, 2015; Purnomo et al.,

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2021). Two most commonly used recycle aggregate, i.e., recycled concrete or paving-originated aggregates, supports the sustainability of the aggregate's lifecycle. The main issue when dealing with recycled aggregates is the quality: the physical and mechanical properties that are highly influenced by the original binding agent residue. The quality and origin of cement highly influence the alteration of the aggregate properties when dealing with concrete and mortar. For asphalt pavements, the asphalt is the main affecting material. The untraceable origin and the wide range of cement and asphalts make the aggregates a very unreliable material for recycling purposes.

The use of recycled asphalt aggregate (RAA) in mortar has been widely studied (Abraham & Ransinchung, 2018; Sola & Ozyazgan, 2019; Debbarma et al., 2020; Shi et al., 2020). In general, RAA from road scarifying is used directly as a constituent in mortar. The studies concluded that the use of RAA in mortar decreases the mechanical properties as a function of asphalt content in the RAA. The main source of this depreciation is the poor bond. Debbarma et al. (2020) recommend applying surface treatments (mechanically or chemically) to remove the residue from the surface to stabilize the aggregate. The following is an overview of methods nowadays used.

The use of microwaves for concrete aggregate recycling started as early as 2011 and is still being investigated to date (Akbarnezhad et al., 2011; Choi et al., 2014; Mousa et al., 2020; Wei et al., 2021a; 2021b; 2021c). Microwave heating is also used in asphalt road maintenance work, such as hot in-place recycling. This method does not completely remove asphalt from the aggregate surface but only softens the asphalt to facilitate mixing of the recycled asphalt pavement with additional asphalt or new aggregate (Benedetto & Calvi, 2013; Sun & Sheng, 2020; Gulisano & Gallego, 2021). Mechanically removing the thin layer of the bonding agent using acid and lime immersion and mechanical rubbing (Kazmi et al., 2019) was also attempted. Improving the contaminated surface was conducted by accelerated carbonation techniques and slurry wrapping (Wang et al., 2020). The two basic approaches differed in their method: the first was by film removal, and the second was by treatment of the coated surface. Most recent is the introduction of nano-silica technology through a combination of pre-spraying, air-drying, and particle size evaluation (Gao et al., 2020; Li et al., 2021), and the self-healing concept. The latter evolves around re-hydrating, bacterial self-healing, and micro-encapsulation (Li et al., 2021). An overview of methods, their advantages, and disadvantages, are outlined in the following works (Wang et al., 2020; Tam et al., 2021).

This work is focused on the reuse of RAA in mortar. The focus is directed on mortar, since the behavior of mortar represents a wide range of cement-made products such as paving blocks, concrete, and masonry. Three types of aggregate were used: virgin aggregate (VA), recycled asphalt aggregate (RAA), and RAA after removing the thin asphalt film using the pyrolysis method (designated as pyrolysis recycled asphalt aggregate (PRAA)). The Pyrolysis method is a unique new surface treatment method to remove and stabilize the residual asphalt film by heating and is expected to contribute to increasing the mechanical properties of the mortar due to an aggregate-to-mortar bond improvement.

2. Methods

The behavior of mortar-based VA, RAA and PRAA was investigated by studying the mechanical and physical properties of the mortar and the aggregates. The mortar properties evaluated were the compression and tensile strength at age variations of 7, 14, 28, 56 and 180 days; the strength development pattern and progress of the cement's chemical reaction were observed. For virgin aggregate-based mortars, the design strength is customarily obtained at 28 days; the cement hardening process was prolonged, but the increase is fairly moderate. The physical data from the aggregates being investigated were the absorption and abrasion to underline the outcome. The aggregate size was within the range of 2.38 mm to 4.76 mm, and the asphalt percentage (relative to the weight) was 4.5% - 8.0%. The maximum asphalt content in an asphalt mixture is 8% (for open-graded hot mixed asphalt). The thickness of the asphalt layer influences the mechanical behaviour of the aggregates (Dong et al., 2020).

The RAA was processed through a pyrolysis reactor with a temperature of 500o C for 5 hours and transformed into PRAA. During the process in the vacuum chamber, the chemical structure of the asphalt residue was transformed into hydrocarbon gas and a liquid oil substance that could be separated from the aggregates (Jing-Song et al., 2003; Cheng et al., 2017; An et al., 2018). The X-ray fluorescence (XRF) test showed that RAA consists of 18% carbon, while the PRAA has an 83% carbon content by mass. The 65% carbon transformation originated from a chemical degradation of 15%, 31%, 7%, and 6% for Al2O3, SiO2, CaO, and Fe2O3, respectively.

The mortar specimens had an aggregate-controlled environment for the gradation, water content, and organic and inorganic impurities. The specimens were cured by water submersion until one day before testing, then dried. The mix proportion for the aggregates and cement was 2:1 with a water-cement-ratio of 0.5 by mass. The water-cement-factor was within the ranges for the RAA (Chaidachatorn et al., 2019). The 50 x 50 x 50 mm cubes for compression strength testing were in accordance with ASTM C109, while the 40 x 40 x 160 mm specimens for tensile testing were guided by ASTM C348. The data were analyzed and evaluated for correctness using statistics, and the data average was taken to represent each category.

Scanning electron microscope (SEM) readings were conducted to observe the changes in surface structure of the VA, RAA and PRAA aggregates. Peaks, valleys and the presence of surface voids were investigated and measured to detect pre-existing cracks and determine overall surface roughness. Valleys would function as interlocking points for the mortar but could weaken the bond when too deep, while pre-existing cracks promote crack propagation in the mortar under loading.

2.1. Aggregate and Mortar Properties

2.1.1. Compression and Tensile Strength

The VA was subjected to petrographic testing on thin-sliced specimens to determine the granulation, crystallinity, mineral composition and relationship between crystals. These results support the determination of the surface stability of the VA and provide information on possible secondary chemical reactions between the aggregates and the cement components. All of these factors influence the bond behavior between the cement and the aggregates. The aggregate was also evaluated to determine its mechanical properties. For this purpose, 25 x 50 mm cylinders were core-drilled from the bulk stone and subjected to compression stresses until failure. This test resulted in the strength, modulus of elasticity and material behavior.

The mortar cubes were subjected to uniform compression stress. The test was displacement controlled with an increment of 0.004 mm/sec.

Due to disparities in initial stiffness, the tensile strain in the aggregates and cement paste in the X and Z-axis direction differ (Figure 1). The bond between the cement and the aggregate bridges the differentiation in the strains until the tensile bond strength limit. The larger the stiffness modulus dissimilarities, the higher the tensile strains in the interface.

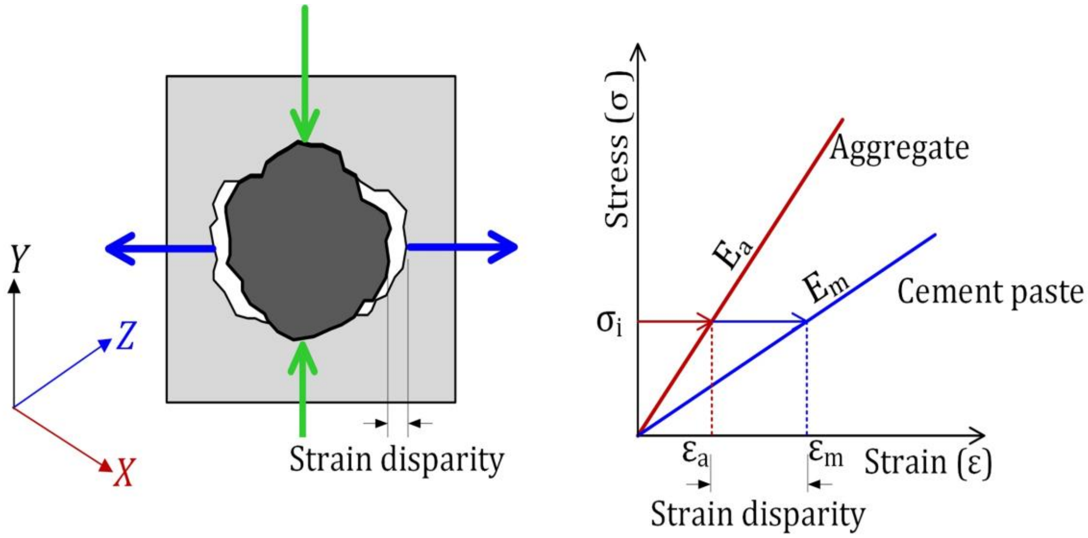


Figure 1 Bond loss due to strain disparities in compression

Failure in the mortar is almost always initiated in the interface between the aggregate and the cement paste (Königsberger et al., 2018; R. Wang et al., 2020; Xu et al., 2021). The strength of the mortar is a direct function of the tensile bond strength between the aggregate and the cement (Shen et al., 2019; X. Wang et al., 2020).

The flexure tests conducted on 40 x 40 x 160 mm specimens resulted in the direct tensile strength of the mortar. The bond holds the differentiation between the tensile strains of the cement and aggregates. The response of the pyrolysis treatment on the bond is contemplated from the compression and tensile test results and the visual analysis of the VA, RAA, and PRAA.

2.1.2. Aggregate Classification, Absorption, and Abrasion

The Los Angeles abrasion test was performed to evaluate the strength of the aggregates' surface, resulting in the cohesion state of the aggregate surface subjected to dynamic impact. The abrasion rate influences the overall bond strength as a composite material with cement (Ahmad et al., 2022; Tugrul-Tunc & Alyamac, 2020). While failure generally occurrs due to bond loss between the cement and the aggregate, poor aggregate surface qualities could lead to a failure in the outer layer of the aggregate (Figure 2).

Another important factor is the aggregates absorption degree. The lower the absorption, the less water is entrapped in the aggregate's voids during the mixing process. The water-saturated in the aggregate pores results in a thin water film surrounding the aggregates. This water film promotes the formation of large, thin crystals in the interface, weakening the bond (Kwan & Li, 2012; Ghasemi et al., 2019; Chen et al., 2021). During the preparation stage, the aggregates were standardized. The VA was oven dried for 24 hours and submerged in water for another 24 hours. The surfaces were dried to create a saturated surface dry (SSD) condition. To avoid deviations in the chemical character of the surface, the RAA and PRAA were not exposed to high temperatures in an oven but sun-dried for a number of days until a weight convergence was reached. The RAA and PRAA were further submerged and dried to an SSD condition.

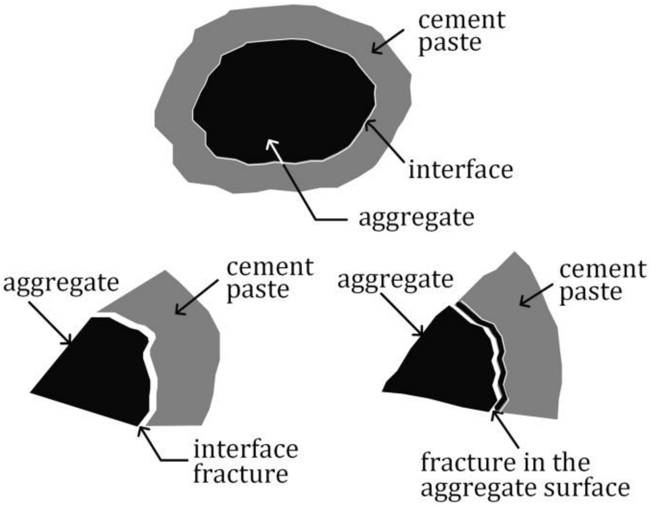


Figure 2 Failure mode in the interface

3. Results and Discussion

3.1. Compression and tensile strength

The aggregates had a compression strength of 125 ~ 145 MPa, a Young's modulus of 65 ~ 70 GPa, and a Poisson's ratio of 0.25. The material behaves linearly until failure. The cement paste had a compressive strength of 55 MPa, an initial modulus of elasticity of 32 GPa, and a Poisson's ratio of 0.22. The aggregate has a much higher compression strength and modulus of elasticity when compared to the cement paste; failure, therefore, will most likely occur in the cement paste or in the interface.

The mortar cubes were tested at several different ages, and the compression strength resulted is as presented in Table 1. The change in strength for every mortar category was compared to the virgin aggregate-based mortar and expressed as the strength ratio.

Table 1 Mortar compression strength ratio to VA

Mortar age (days)	Compression Strength (MPa)				
	VA	RAA	Ratio to VA (%)	PRAA	Ratio to VA (%)
7	33.7	14.4	43%	21.8	65%
14	44.8	19.2	43%	29.0	65%
28	47.6	20.4	43%	33.6	71%
56	49.7	22.3	45%	35.5	71%
180	51.5	23.4	46%	36.6	71%

The data showed that the strength of RAA mortar is substantially lower than the strength of the VA mortar. At all ages, the ratio was 43% ~ 46%. This outcome underlined the findings of (Sola & Ozyazgan, 2017; Brand & Roesler, 2017a; 2017b; Abraham & Ransinchung, 2018; 2019a; Shi et al., 2020), where natural aggregates were gradually substituted by RAA. The findings of (Qiang et al., 2011) suggested that the asphalt film had a negative effect on the cement hydration process within the time frame of 80 hours, however, the data presented in Table 1 demonstrates that this effect had stabilized after 7 days. The pyrolysis procedure improved the performance of the aggregates, and the ratio in compression strength was in the range of 65% ~ 71% for all ages. The visual observation of the fracture plane demonstrated that failure was due to the bond loss between the aggregate and mortar, regardless of the mortar's age, and due to fracture of the outer layer of the aggregates for the RAA mortar (Figure 3). Figure 3a shows the fracture areas in the aggregates' surface and Figure 3b shows the debonding of the interface. Debonding leaves a clean surface, while aggregate surface fracturing leaves a distinguishing mark in the cement paste.

a) Aggregate surface fracture b) Interface debonding

Figure 3 Visual observation of the fracture plane of RAA and PRAA mortar

The compression strength development as a function of mortar age can be seen in Figure 4. For all types of aggregates, convergence in was reached after 28 days. There was no indication of secondary cement hydration. The tensile strength results are presented in Table 2.

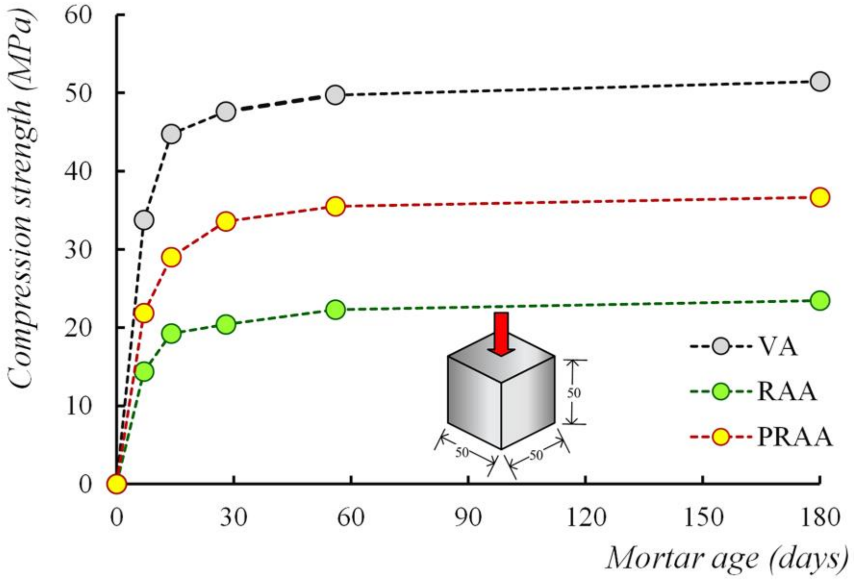


Figure 4 Mortar compression strength development as a function of age

Table 2 Mortar tensile strength ratio to VA

Mortar age (days)	Tensile Strength (MPa)				
	VA	RAA	Ratio to VA (%)	PRAA	Ratio to VA (%)
7	5.0	4.1	83%	4.1	83%
14	7.9	5.1	64%	5.4	68%
28	9.1	5.3	58%	5.9	64%
56	9.5	5.5	58%	6.1	64%
180	9.7	5.6	58%	6.2	64%

The tensile strength ratio declined with age and the pyrolysis procedure had a negligible impact on the tensile strength. Both the RAA and PRAA had a strength ratio of 83% at early ages, declining to 58% and 65% by 180 days, respectively. The tensile strength convergence of all mortars was reached at 28 days, underlining the statement that the RAA and PRAA mortar did not influence the chemical process in the cement (Figure 5).

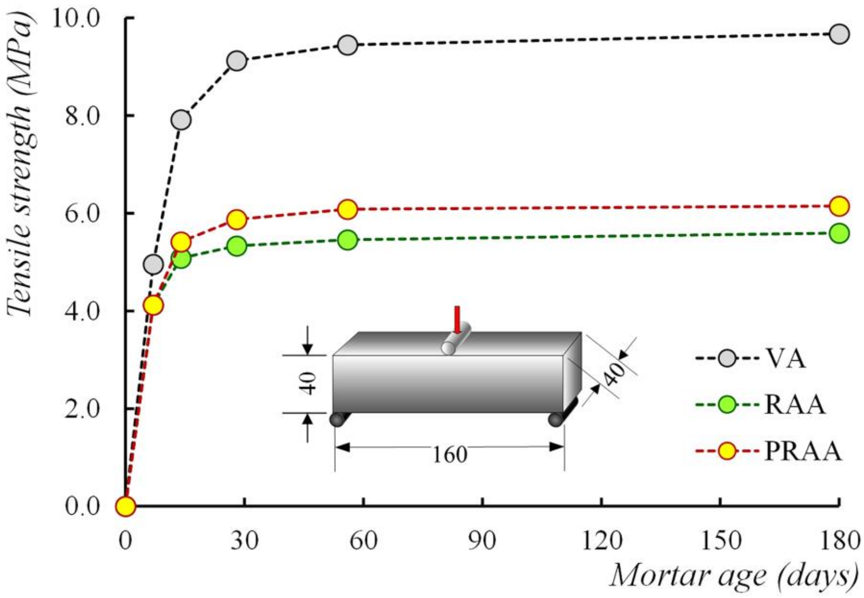


Figure 5 Mortar tensile strength development

The relationship between the mortar compression and tensile strength is generally expressed as: $f_{tr} = C\sqrt{f_c'}$. The suggested coefficient C was 1.3 for the VA, 1.2 for the RAA and 1.0 for the PRAA; where C is the conversion coefficient and f_c' is the concrete compression strength (in MPa). These coefficients slightly overestimate the strength relationship at 7 days but are a very good representation at all ages up to 180 days.

3.2. Aggregate Characteristics and SEM Analyses

Scanning electron microscope analyses is a procedure to visualize a surface using a beam of electrons. The specimen is placed in a vacuum environment preventing reactions between molecules and atoms outside the specimen with the electrons. An electron gun fires the beam to the specimen's surface. The electrons create a range of other electrons, protons, and irradiations depending on the characteristics of the specimen. A combination of lenses is used to read the reflected electrons to form an image interpretable to the eye.

The aggregate is a volcanic rock and considered stable; the minerals do not induce secondary reactions with the cement or water chemical components. In-depth data on the behaviour of andesite as RAA can be found in the work of Zhou et al. (2020).

SEM readings were conducted at 28 days, at a 1000 and 5000-times enlargement. Figure 6 demonstrates the comparison between the surfaces. For the VA, the voids were measured to be four µm, with valleys of six µm, on average (Figure 6a). These valleys were reduced to approximately three µm due to the presence of the asphalt film. The asphalt layer not only created a much smoother surface, but larger voids were also present and more prominently on the surface (Figure 6b). The decrease in strength is also a contribution of the pre-existing micro-cracks on the surface of the asphalt layer. The cracks were evenly distributed and measured at 0.5 µm to 1 µm in width. These cracks promote micro-cracking under loads and accelerate the propagation, leading to premature failure.

The pyrolysis process greatly improved the structural surface (Figure 6c). A rough, uneven surface is seen, improving the bond area and interlocking with the cement paste. However, the toughness and abrasion resistance of the surface is compromised. The results of the Los Angeles testing yielded a 20%, 22%, and 24% abrasion rate for the VA, RAA and PRAA, respectively. From the point of view of aggregate surface conditions, the decrease in both compression and tensile strength of the RAA and PRAA mortar is well explained.

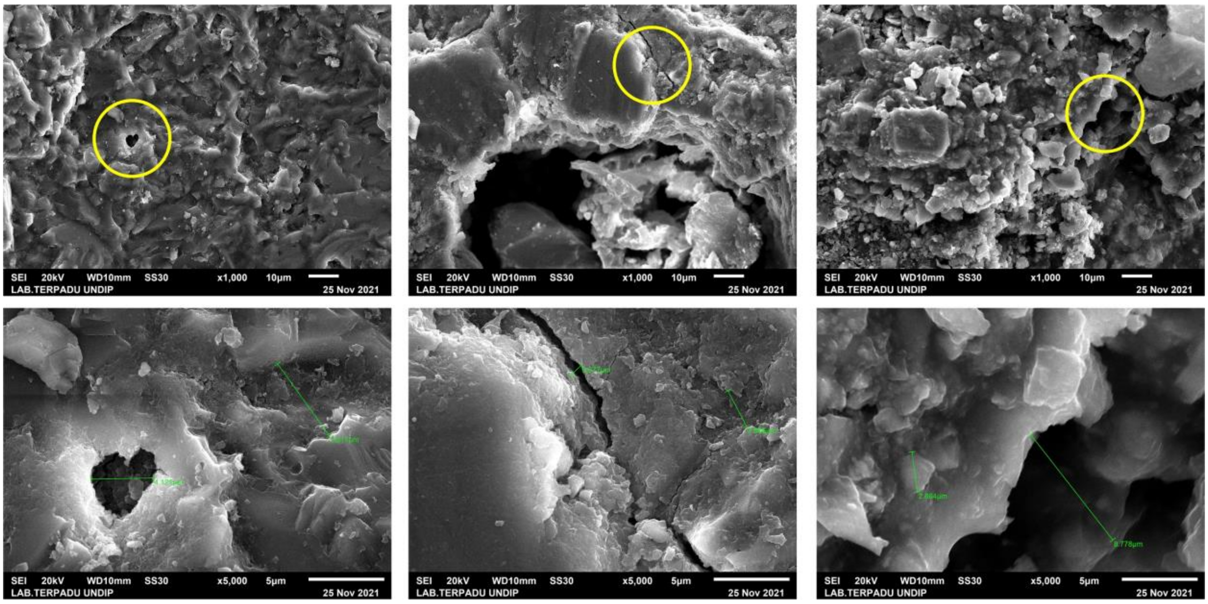

a) VA b) RAA c) PRAA

Figure 6 Surface study on VA, RAA and PRAA

A cross section section reading was performed to study the interconnection among the aggregate, the asphalt layer and the pyrolysis surface, as seen in Figure 7.

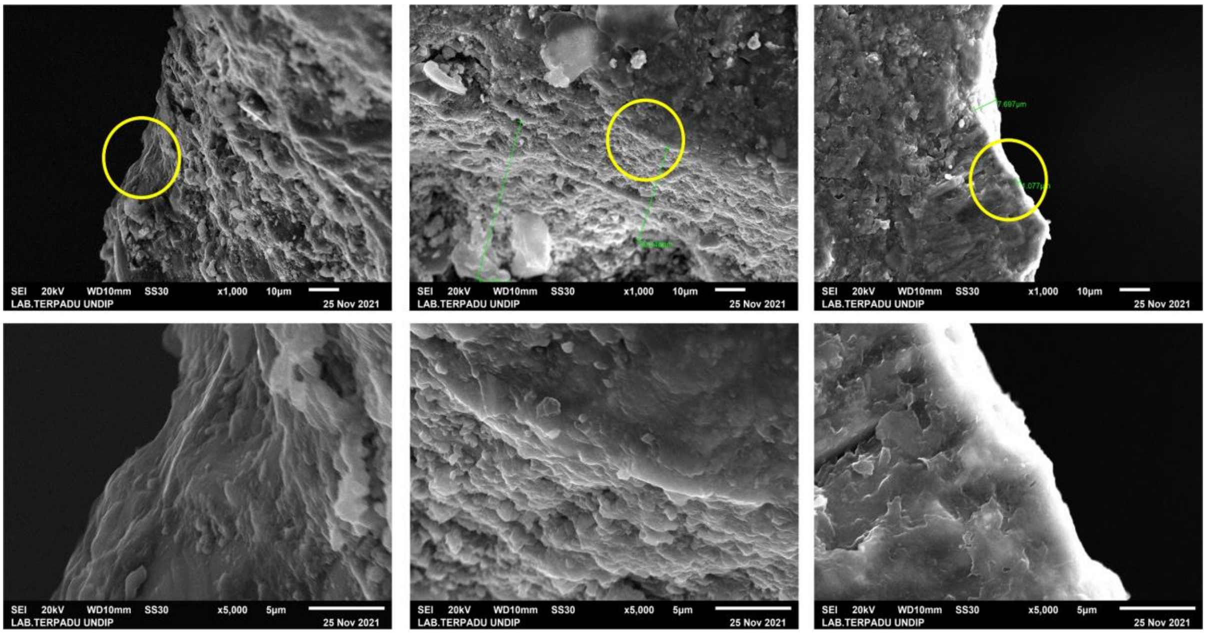
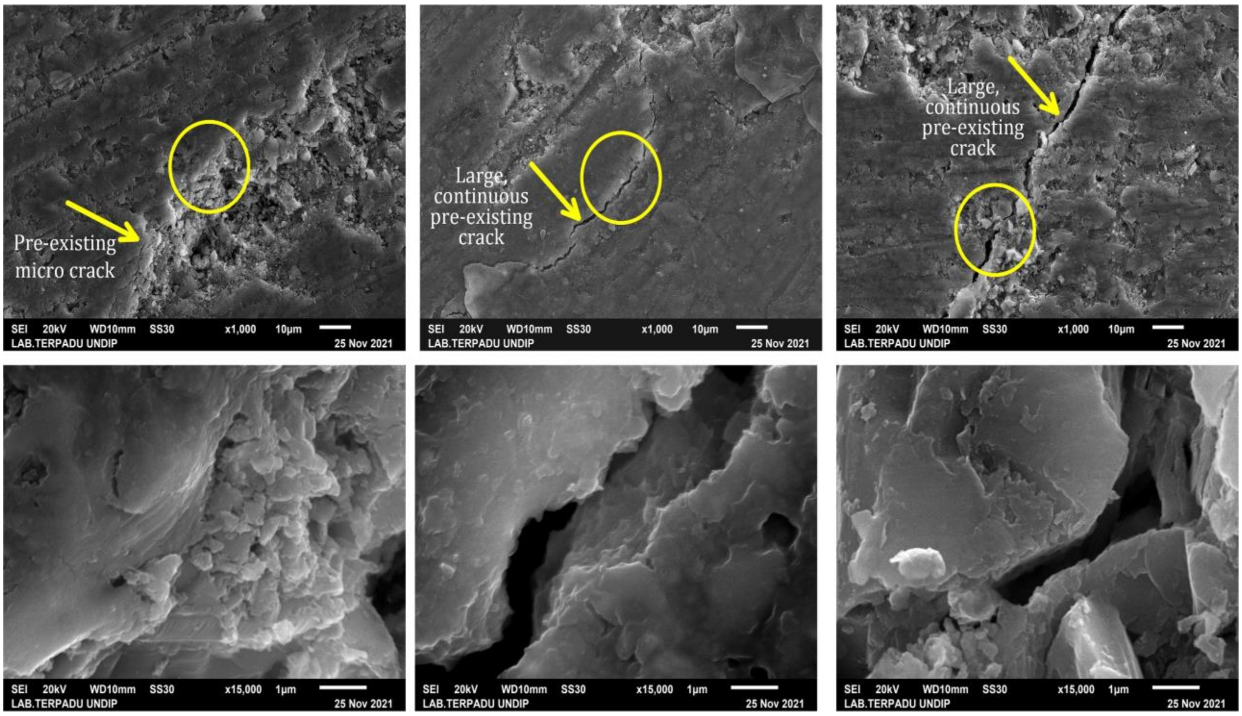

a) VA b) RAA c) PRAA

Figure 7 Cross section study on VA, RAA and PRAA

The VA has a clean surface and no loose particles are detected (Figure 7a). The RAA has an unmistakable asphalt film with a thickness of 30 μm ~ 60 μm. The surface is distinguished by loose, unstable particles, contributing to the poor bond (Figure 7b). Pyrolysis improved the surface and reduced the thickness of the film to 1 μm ~ 7 μm while loose particles were removed completely (Figure 7c). However, the asphalt film cannot remove completely from the RAA surface after heating for five hour and at temperature 500^{o} C.

Finally, visual observations of the aggregate-to-mortar interface are shown in (Figure 8). The specimens were observed at 1000- and 15.000-times enlargement, and not pre-exposed to loading. For all categories, a dense cement paste mass was detected in the vicinity of aggregates with no ettringite formations. The VA has an almost undisturbed interface with a few micro-cracks possibly formed during shrinkage. The cracks were not interconnected, and the rough surface provided a good interlocking between the cement paste and the aggregate (Figure 8a).

The RAA interface has large continuous pre-existing cracks, 0.7 µm ~ 1.0 µm in width. The cracks are a result of the occurrence of fatigue in the asphalt material in the past when resisting repetitive traffic loads or a result of the stripping process of the asphalt material to obtain RAA. The width of the crack can vary depending on the hardness characteristics of the aggregate constituents (Tufail et al., 2017). The cement paste was dense, and no distinguishing cement crystals were detected. The smooth oily surface also provided less interlocking (Figure 8b). The PRAA interface has discontinuous but dominant pre-existing cracks, measuring 0.5 µm ~ 2.0 µm in width. The rough PRAA surface provided a good interlocking mechanism between the cement and aggregates (Figure 8c).



a) VA b) RAA c) PRAA

Figure 8 Aggregate cement paste interface

One of the major causes of compressive and tensile strength depreciation of the RAA and PRAA mortar is the presence, dimension, and magnitude of pre-existing cracks. The wider the cracks, the lower the aggregate strength.

The results of the absorption tests revealed that the VA had 1.42% absorption by weight. The RAA had a slightly elevated rate of 1.49%; the asphalt residue created interconnected voids in the outer layers that trapped additional water. Similar findings were observed by studying the water infiltration in RAA concretes (Bittencourt et al., 2021). The pore structure characteristics were studied in great detail, using mercury intrusion porosimetry techniques to explain the pore classification and capillaries in the RAA mortar (Abraham & Ransinchung, 2019b). The PRAA reduced this rate to 0.59%, as seen in Figure

8c. A thin impermeable film resulted from the pyrolysis process restricting the water absorption into the voids of the aggregate.

4. Conclusions

Research on pyrolysis of recycled asphalt aggregates is limited. Most of the work on improving recycled aggregates does not focus on pyrolysis to stabilize the residual film surrounding the aggregates. Comparing the impact of methods in general, recycled aggregates result in a decrease in compressive and tensile strengths due to the residue of previous binding agents and the non-standard quality of the original material, as stated in the majority of previous research works. The attempt to remove the residual film using a broad range of methods showed that the mechanical properties of the new composite using mortar or asphalt as the binder improved, but could never reach the strength of the original virgin aggregates (VA).

The findings in this study showed that, in compression, the decrease was independent of mortar age; the strength reduction was 55% and 30% for the RAA and PRAA mortars. The strength loss of the RAA predominantly originated from the surface conditions: the thickness of asphalt film, smoother surface, pre-existing micro-cracks, which promote crack initiation and propagation under loading, and the decrease in abrasion resistance, in combination with the presence of loose, unstable particles. The PRAA had a better compression strength, but, despite the removal of the asphalt film, a thin layer of residue remained. The PRAA had a relatively high abrasion rate, weakening the bond between the aggregate and the cement. While no preliminary micro-cracks were present in the aggregate surface and the absorption rate was better compared to RAA, substantial pre-existing cracks were detected in the aggregate-to-mortar interface.

In tension, both the RAA and PRAA exhibited a 40% strength decrease, which is slightly fluctuates with age and stabilises at 28 days. The pyrolysis did not substantially affect the tensile behavior due to the presence of pre-existing cracks. The relationship between the tensile and compression strength can be represented by the coefficient C ranging from 1.3 and 1.2 for the VA and RAA, and 1.0 for the PRAA, using the equation $f_{tr} = C\sqrt{f_c'}$ at any age from 14 to 180 days.

The RAA had a 1.49% absorption, but the PRAA had a substantially lower absorption of 0.59%. The pyrolysis stabilized the surface and prevented water intrusion into the aggregate's inner pores.

The pyrolysis method applied on RAA had a number of advantages: the compression strength increased, and the absorption decreased, reducing the formation of a water film surrounding the aggregate during the cement hydration process. The pyrolysis procedure did not have any impact on the tensile strength for both the RAA and PRAA, however, the overall tensile strength decrease was relatively low, with a minimum reduction of 42% in the VA mortar. The pyrolysis procedure offers a solution to rejuvenate the RAA for mortar usage. The reuse of RAA supports the conservation of natural aggregates.

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