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RESEARCH ARTICLE-CIVIL ENGINEERING

Chemical and Microstructural Changes in Reclaimed Asphalt Pavement Aggregates by Pyrolysis

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Abstract

The utilization of reclaimed asphalt pavement (RAP) aggregates as an alternative for rigid pavements is limited. The main objective of this study is to explore and improve the utilization of RAP aggregates as an alternative material for rigid pavement. Specifically, this study focuses on addressing a significant challenge associated with RAP aggregates, which is their poor bond with cementitious binders. The poor bonding results in low compressive and tensile strengths of concrete or mortar. The poor bonding is mainly due to the presence of a thin oily layer of asphalt residue. A proposed method was carried out to reduce the negative impact on the bond between the aggregate and mortar by exposing the RAP aggregates to the pyrolysis process. The research focused on the analyses of the physical and chemical behavior of the aggregates, using the SEM, EDX, and FTIR approaches, as well as reviewing the mortar in both compressive and flexural tensile strength. The pyrolysis affected the physical and mechanical properties positively and the chemical composition of the RAP showed significant changes. The chemical constituents of asphalt attached to RAP aggregates are hydrocarbons. The thin layer of RAP asphalt is the cause of weak bonding, but this layer was altered by the pyrolysis procedure. As a result, water absorption decreased, which had a positive impact on the hydraulic synergy of cement. It is shown that the pyrolyzing RAP improves the compressive strength and flexural tensile strength through modification of the asphalt residue covering the aggregates.

Keywords RAP · Pyrolysis · Chemical · Microstructure

List of Symbols

f'_c	Compressive strength of mortar (MPa)
f_t	Flexural strength of mortar (MPa)
P	Load (N)
A	Cross-sectional area (mm^2)
M	Bending moment (Nmm)
y	Half height of beam (mm)
Ix	Beam inertia (mm^4)

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Abbreviations

RAP	Reclaimed asphalt pavement
PYRO	Pyrolysis aggregate
FTIR	Fourier transform infra-red
EDX	Energy-dispersive X-ray
SEM	Scanning electron microscope
ASTM	American standard testing material
SNI	Standard Nasional Indonesia
MPa	Mega pascal
C	Carbon
Ca	Calcium
C–H	Hydrocarbon
O–H	Hydroxide
C–S	Carbon sulfide
C–O	Carbon monoxide
S	Sulfur
S–S	Disulfide
Si	Silica
Fe	Ferrit

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1 Introduction

Reclaimed asphalt pavement (RAP) is a result of flexible paving striping when new paving layers are constructed [1–5]. RAP is categorized as a waste material and potentially has a negative impact on the environment [6–9]. Conventionally, RAP is utilized as road shoulder filler [10], road sub-base material, or for patching potholes in combination with the use of hot mix [11, 12]. Occasionally, the material is used in concrete mixes [13, 14]. The Indonesian National Database showed that 50,000 m^3 of RAP is annually produced [15]. Reutilization of RAP in flexible paving was attempted through mixing the RAP with liquid asphalt at various concentrations [16–18]. This method benefits the environment and sustainability through recycling [19]. In terms of strength properties, the RAP resulted in a decreased strength in both compression and tension. The source of this negative effect originates from the existing asphalt layer surrounding the aggregates, weakening the bond with the cementitious material or fresh asphalt [20]. Joice [21] reported that shrinkage cracking often occurs in large quantities of RAP concrete mixes. Increasing the amount of RAP in concrete can weaken the strength due to the presence of a thin asphalt layer in the aggregate [13, 22–25]. Qiang [26] reported that emulsified asphalt cement delays the hydration of cement and asphalt membrane, thus negatively affecting the cement bond to RAP aggregates. Since the thin layer of asphalt residue is the origin of the negative impact, removing this layer becomes crucial in improving the physical and mechanical properties of cementitious products using RAP. However, limited research has been conducted in attending this approach. This work deals with the introduction of the pyrolysis method to the physical removal of to this layer.

Pyrolysis is the process of heating materials to separate carbon compounds at high temperatures [27–29] in an airtight environment, to obtain by-products in the form of oil, gas, and material residues [30–36]. Wang et al. [34] reported that the pyrolysis process produces bio asphalt for the rejuvenation of RAP used for asphalt emulsion re-mixing [32, 37, 38]. The pyrolysis process produces bio asphalt oil products that can improve the durability of asphalt mixtures, but the tensile strength and rutting performance (permanent deformation) decreases [39]. The use of bio asphalt oil for the rejuvenation of RAP material serves as a binder, due to its ability to reduce stiffness at existing conditions and increase asphalt bonding to low temperatures. Rejuvenation with the addition of bio asphalt oil can improve the rheological properties of asphalt adhesive binders [32, 40, 41]. The pyrolysis method was used to extract Buton asphalt sand to obtain asphalt oil for further utilization but did not reuse the sand that had become residue during the pyrolysis process [42–44]. Until now, no one has utilized pyrolysis RAP to recycle the aggregate as mortar material. This research aims to determine the effect of pyrolysis heating on the aggregate of RAP material.

2 Research Significance

Recently, sustainability and control of carbon footprints are of major concerns worldwide. RAP is a waste product that not only contaminates soil and ground water but also consumes natural resources. The utilization of RAP in cementitious products will overcome this problem, while also reducing the leaching of asphalt components through solidification. The attempt to reuse RAP in cementitious products such as mortar and concrete were hindered by the sharp decrease in strength properties making the recycling of RAP virtually unfeasible. The source of the drastic reduction in strength originated from the poor bond between the RAP and binding agent due to the presence of a thin layer of asphalt residue. This study attempted to modify this layer to diminish the poor bonding properties of the RAP using pyrolysis. The pyrolysis procedure transforms the asphalt layer into other material that can further be refined as fuel products. In the process, pyrolysis alters the physical performance of RAP into PYRO aggregates. The PYRO aggregates demonstrated promising results when employed as mortar aggregates. The mechanical and physical properties of mortar are significantly improved. The detailed pyrolysis method is described in this work and highlights the property changes that resulted using advanced testing procedures. The method used herein has never been previously studied and could contribute significantly to environmental awareness to overcome the negative impact of RAP waste.

3 Materials and Methods

3.1 Materials

The RAP used is waste with andesite rock type and asphalt penetration specifications of 60/70. The RAP was taken in bulk form and consisted of various aggregate sizes. The pretreatment selected RAP aggregates had a gradation with the provisions of passing 4.75 mm sieve and retaining 2.36 mm sieve. The RAP aggregates were sun-dried to reduce the moisture content of the RAP pores. The mortar was prepared as a mixture of cement and aggregates with water as the hydration component.

3.2 Procedure Pyrolysis

The pyrolysis process uses a series of devices consisting of a heater, reactor, temperature sensor, and condenser (Fig. 1). Before the pyrolysis process begins, the RAP material is put

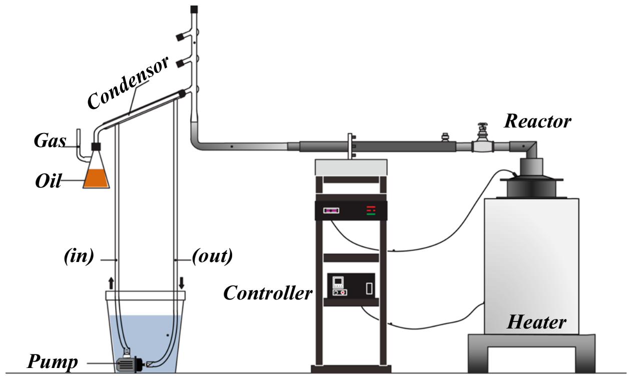

Fig. 1 Pyrolysis process flow

into a reactor with a capacity of 20 kg, then the heater is turned on until it reaches a temperature of 500 °C for 4 h [36]. During the pyrolysis process, changes in temperature, time, and the release of hydrocarbon gas were observed until oil was obtained [44]. When finished, the resulting products are oil, gas, and aggregate solids. The aggregate resulting from the pyrolysis of RAP is called PYRO aggregate.

3.3 Chemical and Microstructure Testing Procedures

This research is divided into two stages, namely, testing the chemical microstructure and mechanical properties of mortar. In the first stage, the RAP and PYRO aggregates were compared to observe the effect of chemical functional groups by means of the Fourier transform infrared (FTIR) method. Meanwhile, to determine the chemical composition of the microstructure, the energy-dispersive X-ray (EDX) and scanning electron microscope (SEM) were used. The analysis of functional groups and chemical microstructure used the 0.074 mm sized aggregates based on the ASTM E986-04 [45]. FTIR testing focused on observing hydrocarbon and carbon sulfide groups, the microstructure was observed at 1000X magnification and the chemical elements formed during the pyrolysis heating were mapped [46, 47]. Physical tests of aggregates included specific gravity [48], absorption, moisture content [49], asphalt content [50], and abrasion [51].

The analysis includes observation of phenomena that occur during the pyrolysis process. Chemical analysis was carried out by reading the functional groups of chemical compounds that react due to pyrolysis (FTIR) using the Perkin–Elmer UATR Spectrum Two tool. Analysis of compound functional groups correlates with analysis of changes in chemical composition (EDX), asphalt content, absorption, abrasion, and aggregate surface morphology (SEM) using the JEOL JSM-6510LA apparatus.

3.4 Mechanical Property Testing Procedure

The second stage of the mechanical properties testing procedure refers to the ASTM C109 [52, 53] standard for mortar compressive strength and ASTM C348 [54, 55] for mortar flexural tensile strength. The mortar mix design has a weight ratio of cement to aggregates were 1:2 and a water to cement ratio of 0.485 water. The mortar specimens were cubes with dimensions of 50 × 50 × 50 mm and beams with dimensions of 40 × 40 × 160 mm.

The compressive strength of mortar can be calculated using the Eq. (1)

$$f_c' = \frac{P}{A} \tag{1}$$

The flexural tensile strength of mortar can be calculated using the Eq. (2)

$$f_t = \frac{M \cdot y}{I_x} \tag{2}$$

In the above equations, f_c' and f_t are the compression strength and modulus of rupture, respectively, P is the axial concentrated load (N), A is the area (mm^2), M is the flexural ultimate moment (Nmm), I_x is the moment of inertia of the section (mm^4), and y is the distance of the centroid to the extreme fibers in tension. The mortar was tested at age 7, 28, and 56 days. The apparatus to test compressive strength and flexural strength was a computer control servo hydraulic concrete press testing machine. Furthermore, analyses were conducted to obtain the compression strength and modulus of rupture as a function of mortar age.

4 Results and Discussion

4.1 Effect of Pyrolysis on Aggregate Properties

The effect of high heating causes the asphalt coating on the RAP aggregates to melt and separate the aggregates from one to another. The reaction of the asphalt layer that occurs in the pyrolysis process has three phases:

1. At a temperature of 200–380 °C, the asphalt in the RAP layer melts.
2. At a temperature of 380–500 °C, the melted asphalt undergoes vaporization in the form of hydrocarbon gases [30, 44].
3. At temperatures above 500 °C, asphalt in the form of hydrocarbon gas flows into the condensation chamber and undergoes a reaction to condense into oil and some LPG gas [43].

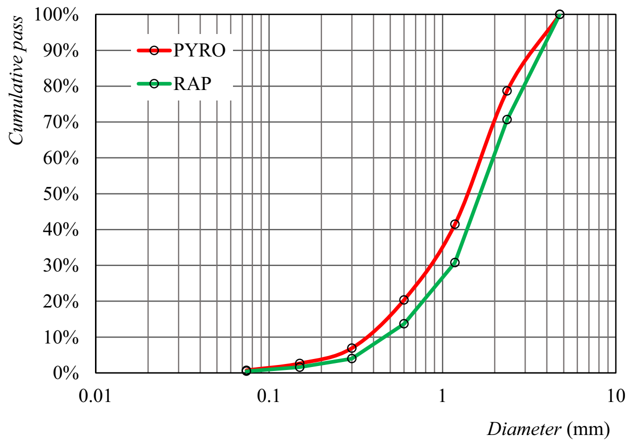

Fig. 2 Change in gradation of PYRO aggregate

This heating process transforms the asphalt that initially has carbon chain bonds above C_{20} into short carbon chains C_5–C_{16} through oxidation [29]. The C_1–C_5 chain is the liquified petroleum gas (LPG) fraction [56] and C_6–C_{16} is the fuel oil fraction [44]. Due to pyrolysis, the RAP aggregate experienced a change in aggregate gradation and is classified as PYRO. Figure 2 demonstrates the fluctuation in gradation of the RAP to PYRO and shows that the PYRO has a much finer constitution.

The physical transformation of RAP aggregate into PYRO is depicted in Fig. 3 where pyrolysis at temperatures above 500 °C causes the release of fine aggregate grains from binding with the asphalt. The amount of aggregate weight released indicates that the asphalt binding phase has ended because hydrothermal reactions with high temperatures can force the asphalt stiffness to change into other forms such as oil and gas [57].

Pyrolysis heating also affects the density of PYRO aggregates. The tests results showed that PYRO had a higher specific gravity with a value of 2.53 and bulk density of 1.52 compared to RAP aggregates (Table 1). PYRO aggregates become more dense due to the high temperature which allows asphalt residue (carbon–sulfur crusts) to enter the pores of the aggregate.

The aggregates were further tested to obtain their toughness [51]. The abrasion of PYRO aggregates is 23.86% higher than that of RAP aggregates. The rationale behind this increase is the fact that above 200 °C microcracks were formed, resulting in a higher fracture degree [58].

Pyrolysis affects the water absorption of aggregates as seen in Table 1. The RAP had an absorption of 1.49% as compared to 0.59% for PYRO. The pyrolysis procedure resulted in a carbon film that to some degree prevented the water from penetrating into the PYRO aggregates. The reduction in absorption rate, however, could not be directly related to

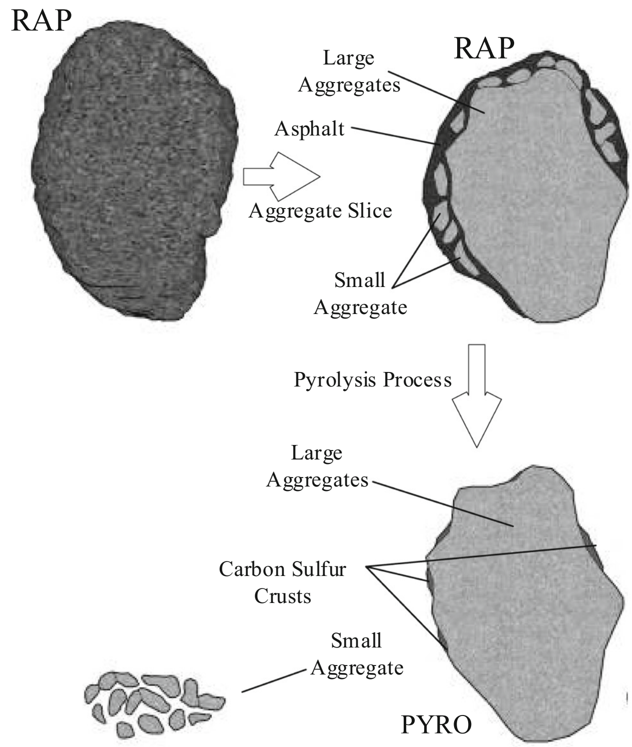

Fig. 3 Transformation of RAP aggregate into PYRO due to pyrolysis

Table 1 Properties of RAP and PYRO comparison

Parameters	RAP	PYRO
Specific gravity	2.26	2.53
Weight content (kg/dm^3)	1.36	1.52
Absorption (%)	1.49	0.59
Abrasion (%)	22.38	23.86

a reduction in voids. This is in line with the findings of Setiadji et al. [20], that the effect of pyrolysis can remove asphalt from aggregates and can increase the strength of mortar.

4.2 Microstructure

The morphology of the material observed at 1000× magnification shows clumps of particles forming grains on the aggregate surface. Three chemical constituents were detected: silica (green), sulfur (red), and calcium (blue), which are shown overlapping on the SEM–EDX interpretation (Fig. 4). Calcium and silica are the main constituents of the aggregate and sulfur indicates the presence of asphalt on the surface of the RAP aggregate (Fig. 4a). The presence of sulfur on the aggregate surface (silica) indicates the presence of asphalt, but after pyrolysis, the sulfur is separated from silica and calcium.

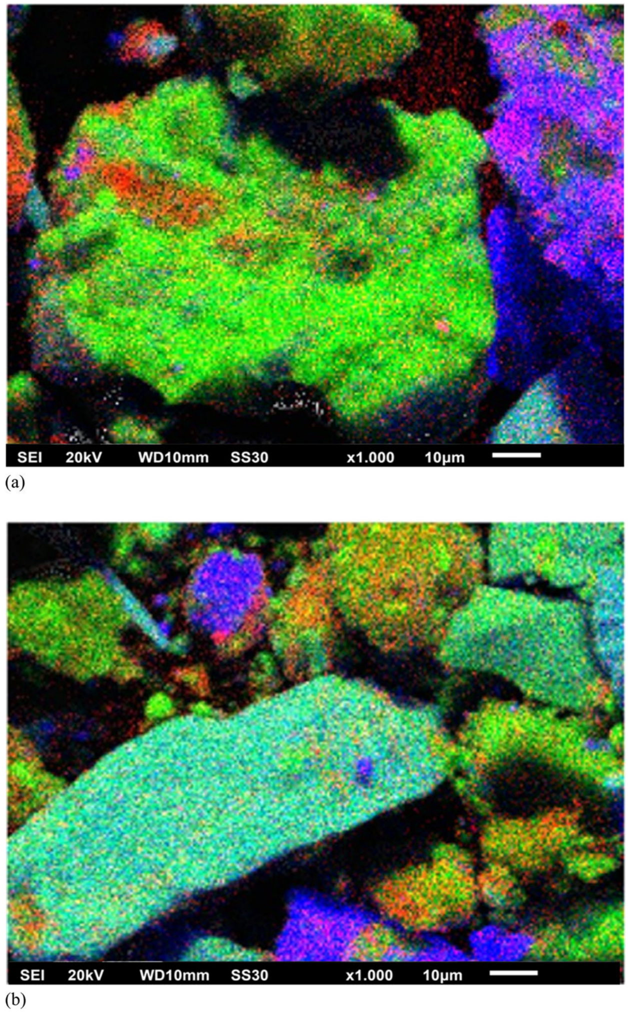

Fig. 4 Mapping element with SEM analysis **a** RAP and **b** PYRO

Table 2 Asphalt content

Parameters	RAP	PYRO	Difference
Asphalt content (%)	4.33	0.17	− 4.16

The PYRO material was part of the aggregate and the asphalt combustion residue (carbon sulfur) shown in Fig. 4b appeared relatively smaller in dimension compared to before pyrolysis (Fig. 4a). The effect of high heating causes [59] the chemical elements of asphalt to break down at the carbon chain. This proves that the pyrolysis effect causes the release of elemental bonds with each other and forms smaller grains [44].

4.3 Effect of Pyrolysis on Aggregate Chemistry

The asphalt content in RAP and PYRO is presented in Table 2. The asphalt content in PYRO has been reduced by 96.07% due to the pyrolysis process. The aggregate composition of RAP and PYRO also always contains oxide compounds. The test results are presented in Table 3. The oxide compounds that increased were Al_2O_3, CaO, SO_3, FeO, MgO, and TiO_2. The chemical elements that dominate asphalt are carbon and sulfur. Pyrolysis RAP heating reaction sequence is the drying process, the oxidation, and cracking reaction. The oxidation and cracking reaction occur in the thin layer of asphalt resulting in hydrocarbon compounds [60] both in the liquid and gas form. After the pyrolysis process is complete, it produces carbon–sulfur (C–S) and disulfide (S–S) compounds in the form of carbon crust on the surface of PYRO aggregates. Calcium and magnesium elements can potentially promote good bonding with cement [61]. This can be proven by the strength analysis of RAP and PYRO aggregate mortar in Figs. 6 and 7.

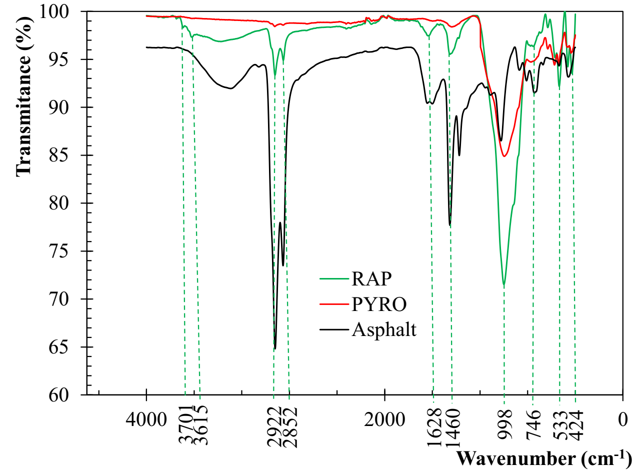

Fig. 5 FTIR RAP, PYRO, and asphalt

The FTIR test results shown in Fig. 5 indicate that the PYRO aggregate (red) is free of asphalt content due to the pyrolysis process. The results of FTIR analysis of PYRO showed transmittance only in the fingerprint and aromatic regions at 400–1000 cm^{-1} reading SiO_2, C–S, and S–S compounds. RAP and asphalt showed identical functional groups in the same wavelength range of transmittance percentage (400–4000 cm^{-1}). The compounds included in these functional groups include hydroxide (O–H), hydrocarbon (C–H), carbon monoxide (C–O), silicon dioxide (SiO_2), C–S, and S–S.

In the wavelength range of 1000–4000 cm^1, PYRO aggregates do not have O–H, C–H, or C–O groups. This proves that the effect of high heating forces the carbon chain compounds to vaporize and react to become oil again during condensation. The pyrolysis reaction changes the asphalt's long (C_{20}) carbon chain bonds into short carbon chains (C_5–C_{16}) and can even remove asphalt from the aggregate [44]. The wave number absorption in the RAP aggregate is in the range of

Table 3 Chemical composition on EDX test

Components	Weight percentage (%)			Difference RAP to PYRO
	Asphalt	RAP	PYRO	
C	82.74	39.24	36.12	− 3.12
SiO_2	4.20	33.38	29.72	− 3.66
Al_2O_3	1.96	9.18	10.13	0.95
CaO	1.87	7.28	9.76	2.48
K_2O	0.22	2.02	0.96	− 1.06
SO_3	5.99	1.12	1.34	0.22
TiO_2	–	0.36	0.47	0.11
MgO	0.17	1.56	4.58	3.02
FeO	2.25	4.16	5.23	1.07
Na_2O	0.59	1.71	1.2	− 0.51

3615–3701 cm^{-1}, which indicates the presence of hydroxide functional groups (O–H). This means that there is still water in the aggregate, while the asphalt shows a liquid state in which there are hydroxide elements in carbon chain bonds. The hydrocarbon compound functional groups at wave numbers 2852–2922 cm^{-1} and 1460 cm^{-1} detected only in the RAP aggregate are carbon chain compounds in the form of asphalt that is still attached to the aggregate. The asphalt content in the RAP aggregate has a content of 4.33% higher than PYRO.

The C–O group at wave number 1628 cm^{-1} is also characteristic of the asphalt carbon monoxide chain, so only the RAP aggregate was identified as still containing asphalt. FTIR testing shows fingerprint areas, namely, the SiO_2 group in the 998–1002 cm^{-1} region is dominant in RAP aggregates. The effect of pyrolysis on the SiO_2 group can slightly reduce the absorption of the compound, thus increasing its transmittance in PYRO aggregates. The functional groups in the aromatic region 424–746 cm^{-1} are C–S and S–S absorptions that identify the chemical bonds of SO_3 in the EDX test composition. Sulfur compounds on RAP aggregates are composed of asphalt bonds, while sulfur on the surface of PYRO aggregates is in the form of carbon–sulfur crusts. This is evidence that residual burning of the asphalt layer that once adhered to the aggregate has occurred and closed its pores. The surface of the PYRO aggregate that is free of asphalt can be reused for concrete mixes and it is suspected that the formation of carbon–sulfur crusts can include calcium and magnesium elements that can promote good bonding with the cement matrix.

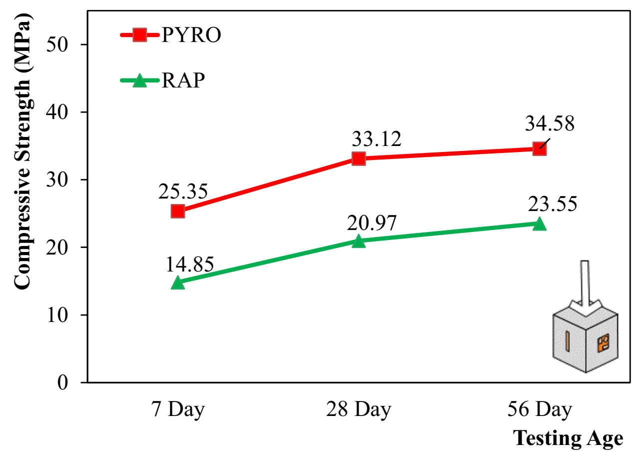

Fig. 6 Compressive strength test for mortar

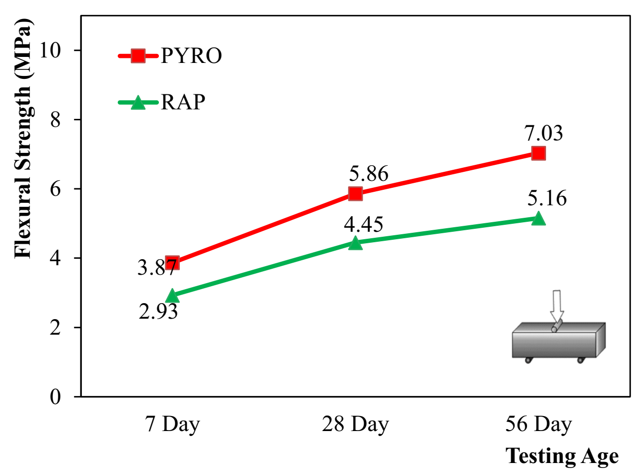

Fig. 7 Flexural test of mortar

4.4 Compressive Test and Flexural Test Results

The increase in compressive strength (Fig. 6) and flexural strength (Fig. 7) of PYRO mortar over RAP mortar is due to several factors including: aggregate, cement matrix reaction, hydration, and interfacial transition zone. The PYRO aggregate had lost its asphalt layer due to pyrolysis heating which caused the specific gravity of PYRO aggregate to be greater than that of RAP. The effect of the cement matrix reacting with the surface of the PYRO aggregates which increased CaO, MgO, and FeO and decreased carbon had an impact on the strength of the mortar. The hydration reaction factor of the cement in the RAP aggregate affected the strength reduction due to the aggregate voids storing easily hydrated water grains, while the PYRO aggregate had lower water absorption. RAP mortars have a brittle asphalt adhesion causing poor cohesive properties between the interface layer of RAP aggregate and cement paste [62].

The chemical hydration reaction of the cement matrix with the aggregate is also influenced by the hardening time, thus affecting the compressive strength and flexural strength values of the mortar to increase with age. The increase in compressive strength of PYRO at 7, 28, and 56 days by 71%, 58%, and 47% against RAP indicates that the hydration of cement in PYRO aggregate is very good, where low absorption can affect the strength of PYRO mortar [20]. In line with Reinhart's [63] theory about the degree of hydration reaction in normal concrete, the change in compressive strength is more gradual than flexural strength. This means that the increase in flexural strength is faster due to the chemical hydration reaction in the PYRO mortar.

The weakness of RAP mortar in the long micro-cracks affecting the mortar structure, is a potential source of RAP aggregate crack propagation leading to structural failure with cement bonding. Due to the occurrence of early cracking as a starting point for structural collapse of RAP mortar [20], it is necessary to remove the asphalt layer on RAP aggregates by pyrolysis. The effect of high heating causes the aggregate to force the residual burning asphalt to fill the pores until it is solid, and this affects the stiffness of the PYRO aggregate.

The higher strength value of PYRO mortar than RAP indicates that PYRO mortar can withstand the load better because it is supported by a solid bond between the aggregate and cement paste. The compressive strength of 33.12 MPa and flexural strength of 5.86 MPa of PYRO mortar can meet the SNI-4857-2017 standard for rigid pavement construction [64]. The analysis in Fig. 8 demonstrates that the improvement of PYRO mortar quality is better and tends to stabilize its flexural strength compared to its compressive strength [65].

5 Conclusion

The reuse of RAP as aggregates in cementitious materials is constrained by the presence of a thin film of asphalt residue that creates a poor bond with the binding agent, negatively influencing the mechanical properties of the final material.

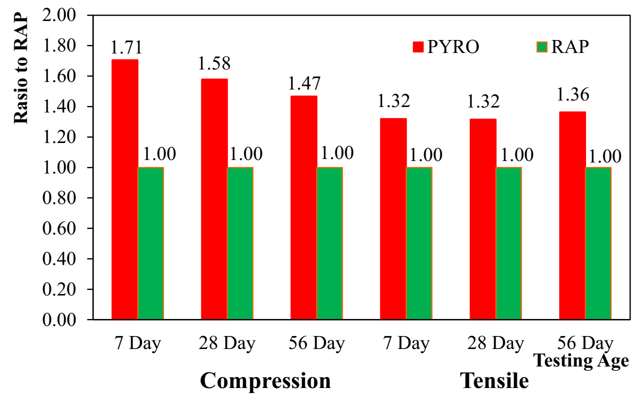

Fig. 8 Improvement of PYRO mortar against RAP

The application of the pyrolysis procedure transforms this layer into carbon–sulfur resulting in an improved bond performance at the interface. Based on FTIR analysis, the effect of pyrolysis causes the residual asphalt layer surrounding the aggregates, predominantly represented by carbon elements, to oxidize. The effect of pyrolysis on morphology shows that this sulfur element is separated and colonized to form new granules called carbon–sulfur. The carbon–sulfur forms a crust surrounding the aggregates that has a positive impact on the bond. The procedure improves the 28-day compression and tensile strength of mortar to 58% and 32%, respectively. It was shown that pyrolysis is more effective in enhancing the compression strength since the resulting carbon–sulfur provided a denser aggregate with a lower absorption rate, and therefore reduces the bleeding surrounding the aggregates.

The study demonstrated that the compression and tensile strength of PYRO mortar at the age of 56 days increased with respect to the 28-day strengths, contradictive to conventional aggregates mortar that reaches convergence at 28 days. The PYRO mortar has a prolonged hardening time, suggesting that the carbon–sulfur positively influences the performance of the cement's hydration process.

The pyrolysis enables the transformation of waste in the form of RAP into reusable and recyclable materials. The by-product of pyrolysis is a crude oil that can be reused in the industry through refinery into gasoline or kerosine. The reuse of the RAP aggregates diminishes the use of virgin aggregates available in nature, supporting the sustainability of natural products.

6 Future Research

This study showed that the utilization of the pyrolysis method to improve the bond in the interface between RAP and mortar significantly enhanced the mechanical properties of mortar. The influence of temperature variation and length of the pyrolysis procedure must be investigated in depth for optimization purposes. An analysis of the best combination between pyrolysis time and temperature must be sought for, and the chemical structure of the remaining layer identified as a function of these factors. Other physical RAP properties that could affect the effectiveness of pyrolysis are the aggregate size, gradation, and the ratio of RAP aggregate's volume-to-area ratio. These are factors that could potentially alter the bond behavior and the variables should be investigated. Furthermore, variations in the chemical structure of original aggregates could result in secondary chemical reactions induced by the alteration of the oily layer into carbon–sulfur. Continuing research should focus on a broader spectrum of asphalt types, especially since the effectiveness of the pyrolysis in removing the asphalt residue is a function of the asphalt properties. Long-term effects such as creep and shrinkage need to be ascertained to ensure a stable cementitious material. It was also shown that after 56 days the compression and tensile strength improve. Studies need to be conducted to determine the convergence age for the PYRO-based cementitious materials as function of age, since the hardening behavior is altered from the 28-day conventional mortar.

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Author Contributions MQ proposing ideas, formal analysis, and writing–review and editing and original draft. HAL contributed to conceptualization, writing, supervising methods, and manuscript checking. Purwanto helped in resources, investigation, and visualization. Widayat contributed to conceptualization, methodology, validation, and manuscript checking. All authors read and approved the final manuscript.

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Data Availability Data sets generated during the current study are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest The authors declare no conflicts of interest.

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