Recycled asphalt pavement – fly ash geopolymer as a sustainable stabilized pavement material

S Horpibulsuk1, M Hoy2, P Witchayaphong3, R Rachan4 and A Arulrajah5

1 Professor and Chair, School of Civil Engineering, and Director, Center of Excellence in Innovation for sustainable Infrastructure Development, Suranaree University of Technology, Thailand
2 PhD Scholar, School of Civil Engineering, Suranaree University of Technology, Thailand
3 Researcher, Center of Excellence in Innovation for Sustainable Infrastructure Development, Suranaree University of Technology, Thailand
4 Department of Civil Engineering, Mahanakorn University of Technology, Thailand
5 Professor, Department of Civil and Construction Engineering, Swinburne University of Technology, Australia

E-mail: suksun@g.sut.ac.th

Abstract. Strength, durability, microstructure and leachate characteristics of Recycled Asphalt Pavement and Fly Ash (RAP-FA) geopolymers and RAP-FA blends as a sustainable pavement material are evaluated in this paper. The strength development of the stabilized materials with and without effect wetting-drying (w-d) cycles was determined by Unconfined Compression Strength (UCS) test. The mineralogical and microstructural changes of the stabilized material were analyzed by X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The leachability of the heavy metals were measured by Toxicity Characteristic Leaching Procedure (TCLP) and compared with international standard. The results show that both RAP-FA blend and RAP-FA geopolymer increase with increasing the number of w-d cycles (C), reaching its peak at 6 w-d cycles. The XRD and SEM analyses indicate that the strength development of RAP-FA blend occurs due to stimulation of the chemical reaction between the high amount to Calcium in RAP and the high amount of Silica and Alumina in FA leaching to production of Calcium Aluminium (Silicate) Hydrate, while the geopolymerization reaction is observed in RAP-FA geopolymer. For C > 6, the significant macro- and micro-cracks developed during w-d cycles cause strength reduction for both RAP-FA blend and geopolymer. The TCLP results demonstrate that there is no environmental risk for these stabilized materials. Furthermore, FA-geopolymer can reduce the leachability of heavy metal in RAP-FA blend. The outcome from this research confirms the viability of using RAP-FA blend and RAP-FA geopolymer as alternative sustainable pavement materials.

1. Introduction
Recycled Asphalt Pavement (RAP), a combination of aggregate and asphalt binder, is obtained from spent asphalt extracted from roads that have reached the end of their design life [1]. Roads are a central component of many nations’ infrastructure and present a wide array of opportunities for the usage of vast quantities of recycled materials such as RAP. Several researchers [2, 3] reported that...
RAP could be used as pavement material when it was stabilized with cement or chemical stabilization. However, the use of cement leads to negative environmental impacts, which immediately requires alternative green material. Fly Ash (FA) based geopolymer is an environmentally friendly additive, which potentially produce better mechanical properties than Portland cement [4].

The usage of waste by-products in civil infrastructure enables a more durable alternative to quarried materials resulting in conservation of natural resources and decreased energy use. Hence, the durability of the stabilized material under severe climatic condition is a crucial parameter when used in road construction application. Cyclic wetting-drying (w-d) test simulates weather changes over a geological age and is considered to be one of the most appropriate simulation that can induce damage to pavement material [5]. Though the utilization of recycled waste materials in highway construction can be considered as having significant impacts on resource management, the hazardous compounds that can leach out and pollute the water resource should also be considered [6].

Therefore, this research attempts to study the possibility of using a green stabilizer (FA-geopolymer) to improve engineering properties of RAP to be a sustainable pavement material. The engineering properties of RAP-FA blend as a control material are also investigated to examine the effect of geochemical agent. First, the strength development of RAP-FA geopolymer and RAP-FA blend with and without effect of w-d cycles are determined by unconfined compression strength (UCS) test. The mineralogical and microstructural changes before and after the w-d cycles were examined by the application of X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analyses. Finally, an environmental assessment was carried out by the leachate test to estimate the contaminate concentration from the RAP-FA geopolymer and RAP-FA blend and compared with international standard. The outcomes of this research will have significant impact on construction guidelines and specification for using RAP-FA blend and RAP-FA geopolymer in road construction applications.

2. Materials and methods

2.1. Materials

In this research, RAP samples were collected from a mill asphalt pavement stockpile in Nakhon Ratchasima province, Thailand. The gradation and the engineering properties of air-dried RAP are shown in Figure 1 and Table 1, respectively. The chemical and mineral composition of RAP, obtained by X-Ray Fluorescence (XRF) and XRD analyses, are presented in Table 2 and Figure 2, respectively.

FA, obtained from the largest lignite power plant in the northern region of Thailand, was used in this study. The grain size distribution curve of FA, obtained by a laser particle analyzer, is shown in Figure 1. Table 2 summarizes the chemical composition of FA using XRF analysis. The XRD pattern of FA is also shown in Figure 2. Liquid alkaline activator (L) is a mixture of sodium silicate (Na$_2$SO$_3$) and sodium hydroxide (NaOH) solution with a 10 M concentration.

2.2. Sample preparation

The RAP-FA geopolymer is a combination of RAP, FA, and L (NaOH+Na$_2$SiO$_3$). The NaOH/Na$_2$SiO$_3$ ratios studied were 100:0, 90:10, 60:40, and 50:50. The RAP-FA blend, which is a mixture of RAP, FA, and water, was prepared as a control material to investigate the effect of L on strength development. FA replacement ratios are 10%, 20%, and 30% by weight of RAP.

The mixtures were compacted in a cylindrical mold (101.6 mm in diameter and 116.3 mm in height) under the modified Proctor energy [7] to determine the optimum water content (OWC) and optimum liquid alkaline activator content (OLC). The sample at the OWC and OLC were prepared for UCS test following the ASTM D1633 [8]. The samples after 7 and 28 days of curing at room temperature (RT), were soaked in water for 2 hours and then were air-dried for 1 hour prior to UCS test according to the specification of the Department of Highway, Thailand [9].
2.3. Wetting and drying (w-d) test
Standard w-d test method for compacted soil-cement mixture [10] was adopted for this study. 28-day samples were submerged in water for 5 hours then dried in an oven at 70°C for 42 hours and air-dried for 1 hour. This is counted for 1 cycle. The UCS of the samples were measured at 1 to 20 w-d cycles and compared with that of the samples without w-d cycle to investigate the effect of w-d cycles on the UCS.

Table 1. Geotechnical properties of RAP.

| Parameter                  | Values  | ASTM   |
|----------------------------|---------|--------|
| USCS Classification       | SP      | D2487-11 |
| Specific gravity           | 2.70    | D1883-07 |
| CBR (%)                    | 10-15   | D577-12 |
| Water absorption (%)       | 6.80    | -      |
| Swelling ratio (%)         | 0.20    | -      |
| Max. dry unit weight (kN/m³)| 17.50  | D1557-12 |
| Optimum water content (%)  | 4.10    | D1557-12 |

Table 2. Chemical composition of RAP and FA.

| Chemical formula | RAP   | FA    |
|------------------|-------|-------|
| SiO₂             | 3.15  | 40.13 |
| Al₂O₃            | 4.78  | 20.51 |
| FeO₃             | 0.10  | 5.83  |
| CaO              | 41.93 | 12.45 |
| MgO              | 36.18 | 3.11  |
| SO₃              | 0.89  | 0.42  |
| LOI              | -     | 0.40  |
2.4. *Mineralogical and microstructural analyses*

UCS development of the samples before and after w-d cycles test were examined by using XRD and SEM analyses, which performed on the small fragments taken from the broken portion of the UCS samples to investigate the mineralogical and microstructure changes, respectively.

2.5. *Toxicity characteristic leaching procedure (TCLP) test*

The Toxicity Characteristic Leaching Procedure (TCLP) test is the method prescribed by the U.S. EPA guidelines (Method 1311) to determine if the solid waste is hazardous. The TCLP tests were carried out on 100% RAP, RAP-FA blend and geopolymers for different types of heavy metal.

3. *Results and discussion*

3.1. *Unconfined compression strength (UCS)*

Figure 3 summarizes the UCS results of the RAP-FA blend and RAP-FA geopolymers for various NaOH/Na$_2$SiO$_3$ ratios, curing condition (7 and 28 days at RT). It is noted that the UCS values of both RAP-FA blends and geopolymers increase with increasing curing time. This result is similar to that reported in previous research on the strength development of cement-stabilized RAP and fly-treated RAP [3].

The 7-day UCS value of both RAP+20%FA blend and RAP+30%FA blend are greater than the strength requirement specified by the Thailand national road authorities in which UCS > 1,723 kPa and UCS > 2,413 kPa for both low and high volume roads, respectively [9, 11]. The UCS of RAP-FA geopolymer at NaOH/Na$_2$SiO$_3$<10:90 is higher than that of RAP-FA blends and the UCS values of RAP-FA geopolymers increase when NaOH/Na$_2$SiO$_3$ ratio is decreased. However, the UCS of RAP-FA blend and geopolymer improves insignificantly when the FA replacement ratio exceeds 20%, indicating this to be the optimal ratio. Hence, the RAP+20%FA blend and geopolymer samples curing age of 28 days were selected for durability test.

![Figure 3. UCS of RAP-FA blends and RAP-FA geopolymers cured for 7 days and 28 days.](image)

3.2. *Wetting-drying cycled strength*

The UCS of RAP+20%FA blend and RAP+20%FA geopolymer at various number of w-d cycles, C is presented in Figure 4. The UCS of RAP+20%FA blend and RAP+20%FA geopolymer increases with increasing C, up to C = 6 and then decreased when C > 6. Previous research on effect of w-d cycles on strength development of FA stabilized with lime and gypsum indicated the strength increase due to the development of cementitious compounds during w-d process [12]. The UCS of RAP+20%FA geopolymer at NaOH/Na$_2$SiO$_3$ = 100 and 50:50 increases sharply after the first w-d cycle and is much higher than that of RAP+20%FA blend for all C tested. Although the RAP+20%FA geopolymer sample with NaOH/Na$_2$SiO$_3$ = 50:50 possesses higher UCS than the sample with NaOH/Na$_2$SiO$_3$ = 100:0 within the first 3 w-d cycles, it has lower UCS when C > 6.
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Figure 4. Relationship between strength and number of w-d cycles of RAP+20%FA blend and RAP+20%FA geopolymers.

Figure 5. Relationship between weight loss and number of w-d cycles of RAP+20%FA blend and RAP+20%FA geopolymers.

The relationship between the weight loss of RAP+20%FA geopolymer and the RAP+20%FA blend versus number of w-d cycles, C is illustrated in Figure 5. The weight loss of both RAP+20%FA blend and geopolymer remarkably increases within the first w-d cycle and thereafter gradually increase with an increase in C. It is noted that the weight loss of RAP+20%FA geopolymer for NaOH/Na₂SiO₃ ratios of 100:0 and 50:50 is lower than that of RAP+20%FA blend due to the stronger RAP-FA geopolymer bonding structure. The effect of cyclic w-d cycles on the external surface of the RAP+20%FA blend and RAP+20%FA geopolymer at NaOH/Na₂SiO₃ ratios of 100:0 and 50:50 is evident in Figure 6a, b and c, respectively at a particular C = 20.

Large macro-cracks and surface deterioration on the RAP+20%FA blend are clearly observed, which leads to strength loss. On the other hand, Figure 6b obviously shows the minimum cracks on the surface of the RAP+20%FA geopolymer at NaOH/Na₂SiO₃ = 100:0, while more cracks are observed for NaOH/Na₂SiO₃ = 50:50 (Figure 6c). This imply that the samples at NaOH/Na₂SiO₃ = 100:0 has stronger bonding structure than the sample at NaOH/Na₂SiO₃ = 50:50. From the cyclic w-d results and the photos, it is evident that RAP+20%FA blend provides a fairly good durability when subjected to w-d cycles. FA geopolymer can enhance the durability of RAP-FA material, especially for the sample at NaOH/Na₂SiO₃ = 100:0.

3.3. Mineralogical and microstructural changes
The XRD patterns of RAP+20%FA blend at various C are shown in Figure 7. Without w-d cycle (C = 0), the RAP+20%FA blend (Figure 7a) contains the amorphous phases of Calcium Magnesium as the predominant minerals (Silica- and Alumina-products), such as Anorthite, Diopsite, Ladradorite, and Ettringite. These new minerals are formed when RAP is mixed with FA (RAP-FA blend). In other words, the chemical reaction between the high amount of Calcium of RAP results in the formation of

![Figure 4](image-url)
![Figure 5](image-url)
![Figure 6](image-url)

![Figure 7](image-url)
Calcium Silicate Hydrate (C-S-H) and Calcium Aluminate Hydrate (C-A-H), similar to the hydration of Portland cement [13], that can enhance the strength development.

The increase in peaks corresponded to Anorthite, Diopside, and Ladradorite with increasing C to 6 is observed by comparing Figure 7b (C = 1) with Figure 7c (C = 6), that indicates the increase of C-A-S-H. Drying at 70°C for w-d test evidently enhances the cementitious products (C-A-S-H) [14]; i.e., an increased temperature results in a faster moisture diffusivity of the cementitious materials and hence cement hardening [15]. The same is however not true for C > 6. The temperature affects the water physical properties (density and surface tension) [16] and causes the coarsening of the pre structure in relation to Ettringite dissolution and C-S-H alteration [14]. Figure 7d indicates the presence of Ettringite and the decreased intensity of Anorthite and Diopside minerals when the samples are subjected to 12 w-d cycles. Ettringite is a hydrous mineral that exhibits expansive behavior upon wetting [17] and makes the RAP-FA blends potentially volumetrically unstable [18].

Beside the XRD results, SEM images of RAP+20%FA blend at various C are illustrated in Figure 8. The growth of C-A-S-H gel inner and on the spherical surface of FA with increasing C (C = 0 to 6, see Figure 8a-c) is observed while reduction in cementitious gel at the C = 12 (Figure 8d) is detected, which confirms the XRD results.
Figure 9 and 10 present the XRD patterns of RAP+20%FA geopolymer at NaOH/Na₂SiO₃ ratios of 100:0 and 50:50 and at various w-d cycles, respectively. As a result of the alkaline activation, new geopolymerization products inclusive of Albite, Nepheline, and Analcime are observed at the broad hump between 22-33°20 in the XRD patter of the sample at NaOH/Na₂SiO₃ = 100:0 (Figure 9a) and at NaOH/Na₂SiO₃ = 50:50 (Figure 10a) for C = 0. A good performance of geopolymer was observed when the precursor was in contact with the alkali activator at curing temperature of 40–75°C [19]. Therefore, the C-A-S-H from RAP-FA blend co-exist with the geopolymer products and contribute to the additional strength development of RAP+20%FA geopolymer in the first 6 w-d cycles. The silica present in sodium silicate is highly soluble and consequently incorporated immediately into the N-A-S-H gel, hence more geopolymer products are detected in the XRD patterns of the geopolymer sample at NaOH/Na₂SiO₃ = 50:50 (Figure 10b and c) compared with those at NaOH/Na₂SiO₃ = 100:0 (Figure 9b and c). Consequently, the UCS values of RAP+20%FA geopolymer at NaOH/Na₂SiO₃ = 50:50 are higher than those at NaOH/Na₂SiO₃ = 100:0 within the first 6 w-d cycles (see Figure 4).

The SEM images of RAP+20%FA geopolymer at NaOH/Na₂SiO₃ ratios of 100:0 and 50:50 (Figure 11 and 12) were examined to support the XRD results. The geopolymerization products in the sample at NaOH/Na₂SiO₃ = 50:50 are more than those in the samples at NaOH/Na₂SiO₃ = 100:0 for C< 6. For C> 6, a particular C = 12, the micro-cracks are clearly observed in RAP+20%FA geopolymer samples (Fig. 11d and 12d) due to the loss of moisture content resulted in external surface cracks leading to a strength loss.

It is noted that the sample at NaOH/Na₂SiO₃ = 50:50 (Figure 12d) has more micro-cracks than the sample at NaOH/Na₂SiO₃ = 100:0 (Figure 11d). Consequently, the rate of strength reduction of the sample NaOH/Na₂SiO₃ = 50:50 is higher than that of the sample at NaOH/Na₂SiO₃ = 100:0 when C> 6. This indicates that the RAP+20%FA geopolymer with NaOH/Na₂SiO₃ = 100:0 has higher durability against w-d cycles than that with NaOH/Na₂SiO₃ = 50:50. This high durability is attributed to the formation of stable cross-linked alumino-silicate polymer structure.

3.4. Toxicity characteristic leaching procedure results
Table 3 shows the leachate analysis of 100%RAP and RAP+20%FA blends using acetic leachate extraction. According to benchmark mandated by the U.S. Environmental Protection Agency (EPA) for storm-water sampling, pH values should be in the range of 6 to 9 [20].

Leachate results show that pH level in 100%RAP is 5.12 and 5.59 for RAP+20%FA blend, which are within allowable limits. Table 4 presents the prescribed limits for drinking water and the threshold for hazardous waste defined by the U.S. EPA. Wartman et al. [21] reported that a material is designated as a hazardous waste in according to U.S. EPA if any detected metal is present in concentrations > 100 times the drinking water standards. Based on this criterion, the comparison of TCLP results between Tables 3 and 4 indicated that all metal contaminates are within acceptable limits.

From a geotechnical engineering perspective, the research results indicate that RAP is mechanically and economically viable for use in pavement base application when it stabilized with 20% of FA. Besides having a high UCS, the RAP-FA blend exhibits good durability against w-d cycles, which can be attributed to the growth of C-A-S-H. FA geopolymer can significantly improve the strength and durability of RAP-FA blend. Furthermore, these materials provide a positive environmental impact as environmental test results show no significant risk to the ground water or stream water line.
Figure 9. XRD patterns of RAP+20%FA geopolymer (NaOH/Na$_2$SiO$_3$ = 100:0) samples at: (a) $C = 0$, (b) $C = 1$, (c) $C = 6$, and (d) $C = 12$.

Figure 10. XRD patterns of RAP+20%FA geopolymer (NaOH/Na$_2$SiO$_3$ = 50:50) samples at: (a) $C = 0$, (b) $C = 1$, (d) $C = 6$, and (d) $C = 12$.

Figure 11. SEM images of RAP+20%FA geopolymer (NaOH/Na$_2$SiO$_3$ = 100:0) samples at: (a) $C = 0$, (b) $C = 1$, (c) $C = 6$, and (d) $C = 12$.

Figure 12. SEM images of RAP+20%FA geopolymer (NaOH/Na$_2$SiO$_3$ = 50:50) samples at: (a) $C = 0$, (b) $C = 1$, (c) $C = 6$, and (d) $C = 12$. 

Table: XRD patterns and SEM images of RAP+20%FA geopolymer samples.
4. Conclusions
The present study investigated the possibility of using the RAP-FA blend as a sustainable pavement material. The 7-day UCS of the compacted RAP-FA blend at OWC meets the strength requirement for base course specified by national road authorities for both 20% and 30%FA replacement. The UCS improves insignificantly when the FA replacement ratio exceeds 20%, indicating this to be the optimal blend. FA-geopolymer can significantly improve the UCS and durability of RAP-FA blend. The XRD and SEM analyses of RAP-FA blends indicate the growth of C-A-S-H, hence its UCS increase over time. The co-exist of C-A-S-H from RAP-FA blend with the N-A-S-H from RAP-FA geopolymer can enhance the durability of RAP-FA blend remarkably.

The TCLP results indicate that RAP-FA blends and RAP-FA geopolymer can be safely used as a sustainable pavement base application as these materials pose no significant environmental and leaching hazard into soil and ground water sources. In addition, FA geopolymer used can reduce the leachability of metal concentration from RAP-FA blend. This study can confirm the utilization of these recycled materials results in significant energy saving and reduction in greenhouse gas emission.

| Parameter | Sample of acid leachate extraction (mg/L) | 100%RAP | RAP-FA blend | RAP-FA geopolymer (NaOH/Na$_2$SiO$_3$ = 100:0) | RAP-FA geopolymer (NaOH/Na$_2$SiO$_3$ = 50:50) |
|-----------|-----------------------------------------|---------|---------------|---------------------------------|---------------------------------|
| pH        |                                        | 5.12    | 5.59          | 7.59                           | 7.44                           |
| Arsenic   |                                        | <0.01   | <0.01         | BDL                            | BDL                            |
| Cadmium   |                                        | BDL     | BDL           | BDL                            | BDL                            |
| Chromium  |                                        | <0.05   | <0.05         | <0.05                         | <0.05                         |
| Copper    |                                        | BDL     | BDL           | BDL                            | BDL                            |
| Lead      |                                        | BDL     | BDL           | BDL                            | BDL                            |
| Mercury   |                                        | BDL     | BDL           | BDL                            | BDL                            |
| Nickel    |                                        | <0.05   | 0.051         | <0.05                         | <0.05                         |
| Zinc      |                                        | 1.348   | 0.657         | BDL                            | BDL                            |

Note: BDL = Below Detection Limit

Table 4. Comparison of TCLP data analysis with U.S. EPA Requirements.

| Contaminant | Drinking water standard (EPA, 1999) (mg/L) | Threshold for solid waste (EPA, 2009) (mg/L) |
|------------|-------------------------------------------|---------------------------------------------|
| Arsenic    | 0.05                                      | 0.35                                        |
| Barium     | 2.0                                       | 35.0                                        |
| Cadmium    | 0.005                                     | 0.1                                         |
| Chromium   | 0.1                                       | 2.5                                         |
| Lead       | 0.015                                     | 0.5                                         |
| Mercury    | 0.002                                     | 0.05                                        |
| Selenium   | 0.05                                      | 0.5                                         |
| Silver     | 0.05                                      | 5.0                                         |

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