

# **Physico-mechanical, Chemical and Durability Characteristics of Mortar and Concrete Containing Palm Oil Boiler Clinker Aggregate Blended with Rice Husk Ashand Ca-Bentonite**

**Kamolchanok Kueaket**

**A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Engineering in Mining and Materials Engineering Prince of Songkla University**

**2021**

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(Dr.Abideng Hawa)

The Graduate School, Prince of Songkla University, has approved this thesis as partial fulfillment of the requirements for the Master of Engineering Degree in Mining and Materials Engineering.

………………………………………………

(Prof.Dr.Damrongsak Faroongsarng) Dean of Graduate School

This is to certify that the work here submitted is the result of the candidate's own investigations. Due acknowledgement has been made of any assistance received.

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(Assoc.Prof.Dr.Danupon Tonnayopas) Major Advisor

…………………………………..[Signature](http://dict.longdo.com/search/signature)

(Miss Kamolchanok Kueaket) **Candidate** 

I hereby certify that this work has not been accepted in substance for any degree, and is not being currently submitted in candidature for any degree.

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(Miss Kamolchanok Kueaket) Candidate



# **บทคดัย่อ**

งานวิจัยนี้มุ่งศึกษาการนำเอาตะกรันปาล์มน้ำมันมาใช้ประโยชน์ในการแทนที่เป็น มวลรวมธรรมชาติผสมกับเถ้าแกลบและเบนโทไนต์ชนิดแคลเซียม เพื่อผลิตเป็นมอร์ตาร์และ คอนกรีตรักษ์สิ่งแวดล้อม โดยใช้แคลเซียมเบนโทไนต์และเถ้าแกลบแทนที่ปูนซีเมนต์ปอร์ตแลนด์ ประเภท 1 ทั้งสองและสามชนิดที่อัตราส่วนร้อยละ 10 20 และ 30 โดยน้ำหนัก ส่วนผสมทั้งหมด คงที่อัตราส่วนน ้าต่อวัสดุประสานไว้ที่ 0.48 ศึกษาพฤติกรรมเวลาก่อตัวและอุณหภูมิของ ปฏิกิริยาไฮเดรชันของเพสต์ศึกษาโครงทางทางจุลภาคและแร่ประกอบของมอร์ตาร์ด้วยกล้อง จุลทรรศน์อิเล็กตรอนแบบส่องกราดและเทคนิคการเลี้ยวเบนรังสีเอ็กซ์ตัวอย่างมอร์ตาร์ทั้งหมด นำไปบ่มในสารละลายน้ำปูนขาวอิ่มตัวที่อุณหภูมิห้อง 29±3 องศาเซลเซียส ความชื้นสัมพัทธ์ร้อยละ 80-90 เป็นระยะเวลา 7 28 และ 56 วัน ก่อนนำไปทดสอบสมบัติต่างๆ ได้แก่ การดูดซึมน้ำ รูพรุนปรากฏ ก าลังอัด และความคงทนต่อสารละลายกรดซัลฟูริกและโซเดียมซัลเฟต ทดสอบสภาพการซึมผ่านได้

ของประจุคลอไรด์ การดูดซึมน้ำในรูเล็กและกำลังดึงทางอ้อม (แบบบราซิล) ของตัวอย่างคอนกรีต ิจากผลการศึกษา พบว่ามวลรวมตะกรันปาล์มน้ำมันสามารถนำมาใช้แทนที่มวล รวมธรรมชาติเพื่อผลิตมอร์ตาร์และคอนกรีตได้และเมื่อเพิ่มปริมาณวัสดุแทนที่ปูนซีเมนต์ทั้งสองชนิด และสามชนิด ส่งผลให้เวลาก่อตัว อุณหภูมิจากปฏิกิริยาไฮเดรชันของเพสต์ และอัตราการไหลของ มอร์ตาร์สดมีค่าลดลง โดยเวลาก่อตัวและอุณหภูมิปฏิกิริยาไฮเดรชันของเพสต์ผสมเถ้าแกลบร้อยละ 30 มีค่าลดลงร้อยละ 16 และ 14 ตามลำดับ เมื่อเทียบกับสูตรควบคุม ทั้งยังพบว่าอัตราการไหลของ มอร์ตาร์สดในสูตรดังกล่าวมีค่าลดลงถึงร้อยละ 38 เมื่อเทียบกับสูตรควบคุม นอกจากนี้พบว่าในสูตร แทนที่เถ้าแกลบร้อยละ 20 ให้ค่ากำลังอัด 53 เมกะพาสคัล บ่มที่ 56 วัน ในขณะเดียวกัน สมรรถนะความคงทนของมอร์ตาร์ ได้แก่ การดูดซึมน้ำ รูพรุนปรากฏ และความคงทนต่อกรดและ โซเดียมซัลเฟตพัฒนาดีขึ้น เมื่อผสมเถ้าแกลบถึงร้อยละ 30 ที่บ่ม 56 วัน ได้ยับยั้งให้ค่าสูญเสีย น้ำหนักและกำลังอัด เนื่องจากการกัดกร่อนของสารละลายกรดซัลฟิวริกที่ร้อยละ 0.47 และ 1.58

ตามลำดับ เช่นเดียวกับยับยั้งค่าน้ำหนักที่เพิ่มขึ้นน้อยสุดและกำลังอัดที่ลดลงหลังจากแช่สารละลาย โซเดียมซัลเฟตเพียงร้อยละ 0.03 และ 0.32 ตามลำดับ ภาพถ่ายโครงสร้างทางจุลภาคและผลกราฟ การเลี้ยวเบนรังสีเอ็กซ์ของตัวอย่างมอร์ตาร์พบรูปทรงก้อนเกาะกันและเส้นใยของสารประกอบ แคลเซียมซิลิเกตไฮเดรตตกตะกอนหนาแน่นในเนื้อและรูโพรงของมอร์ตาร์สูตรผสมเถ้าแกลบเมื่อ เทียบกับสูตรควบคุม สูตรที่ผสมสองชนิดกับแคลเซียมเบนโทไนต์และสามชนิด จึงส่งผลให้ตัวอย่าง ้มอร์ตาร์สูตรที่เติมเถ้าแกลบมีการพัฒนากำลังอัดและความคงทนได้ดีกว่าสูตรอื่นทั้งผสมสองและ สามชนิด เช่นเดียวกับตัวอย่างคอนกรีตที่ผสมเถ้าแกลบร้อยละ 30 บ่ม 56 วัน มีค่าการซึมผ่านได้ ้ของประจุคลอไรด์ต่ำสุดที่ 336 คูลอมบ์ และค่าสัมประสิทธิ์การดูดซึมน้ำ 0.06 กิโลกรัมต่อตาราง ้มิลลิเมตรต่อรากที่สองของชั่วโมง นอกจากนี้พบว่าค่ากำลังดึงทางอ้อมแบบบราซิลได้พัฒนาเพิ่มขึ้น เมื่อตัวอย่างคอนกรีตมีอายุบ่มมากขึ้น และสูตรแทนที่ด้วยเถ้าแกลบร้อยละ 10 ให้ค่ากำลังดึงสูงสุดที่ 3.80 เมกะพาสคัล บ่ม 56 วัน ดังนั้นเมื่อพิจารณาจากผลการศึกษาดังกล่าวและคำนึงถึงการนำเอา ของเสียมาใช้ประโยชน์สูงสุด นั้นก็คือสมรรถนะของมอร์ตาร์และคอนกรีตใส่มวลรวมตะกรันปาล์มน้ำมัน สามารถปรับปรุงดีขึ้นในสูตรที่ผสมสองชนิดกับแคลเซียมเบนโทไนต์ที่ร้อยละ 10 และเถ้าแกลบที่ ร้อยละ 30 และสูตรผสมทั้งสามที่แคลเซียมเบนโทไนต์ร้อยละ 5-10 และเถ้าแกลบร้อยละ 10-20

ี **คำสำคัญ**: ตะกรันปาล์มน้ำมัน, แคลเซียมเบนโทไนต์, เถ้าแกลบ, กำลังอัด, ความคงทนต่อกรด และซัลเฟต, การซึมผ่านได้ของประจุคลอไรด์



## **ABSTRACT**

This research focused on utilization of palm oil boiler clinker (POBC) as a whole natural aggregate substitution blended with calcium bentonite (CB) and rice husk ash (RHA) for green mortar and concrete productions. The CB and RHA were partially replaced by weight of ordinary Portland cement (OPC), type 1 in three proportions of 10%, 20%, and 30% for binary and ternary blends. All mixtures were taken constant water to binder ratio of 0.48. The behavior of fresh pastes on Vicat setting times and hydration temperature were investigated. The microstructure and mineral phase compositions of mortars were characterized via scanning electron microscope (SEM) and X-ray diffraction technique (XRD). All mortar specimens were cured in lime-saturated water at ambient temperature of 29  $\pm$  3 °C, RH =80-90%for periods of 7, 28, and 56 days before each testing. The harden specimens were casted for examination in these following properties: water absorption, apparent porosity, compressive strength, sulfuric acid and sodium sulfate resistance. The cylindrical concrete specimens were prepared for rapid chloride ion permeability test (RCPT), capillary water absorption, and indirect tensile strength test (Brazilian test).

As a result, it was observed that the POBC aggregate can be potentially substituted natural aggregate for mortar and concrete productions. The setting times, hydration temperature of pastes, and percentage flow of fresh mortars were reduced with an increase in binary and ternary blends. The maximum reduction in setting time and hydration temperature was 16% and 14% for the mix incorporating up to 30% RHA. Consequently, an increase in RHA up to 30% decreased the percentage flow by 38%. Moreover, the compressive strength decreased as an increase in ternary blends with prolonged curing ages, the 56-day highest compressive strength was 53 MPa for the mix incorporating 20% RHA. The durability performances of mortar including water absorption and porosity, sulfuric acid and sodium sulfate resistance improved with an increase in RHA replacement level up to 30% and up to 56-day curing ages. The lowest deteriorating due to sulfuric acid attack was suppressed 0.47% loss in weight and 1.58% loss in compressive strength for the mix incorporating 30% RHA cured for 56 days, as well as the lowest percentage gain in weight and loss in compressive strength due to sodium sulfate attack was 0.03% and 0.32%. The SEM microstructure and XRD patterns of dominant mortars specimens indicated that flocslike and fibrous- like forms of the C-S-H densely formed in the matrix and filled up the pores of the mortar containing RHA compared to that of control mix, binary and ternary mixes incorporating CB. This findings contributed to the strength and durability improvement. Likewise, an increase in RHA replacement level and curing ages reduced the chloride ion penetrability and capillary absorption coefficient of the concretes containing POBC as a coarse aggregate, while an increase in CB increased those one. The lowest chloride ion penetrability and capillary absorption coefficient was 336 coulombs and 0.06 kg/mm $^2/\mathsf{h}^{0.5}$  for the mix incorporating up to 30% RHA at the age of 56 days. The indirect tensile strength of concrete increased with curing ages. The highest indirect tensile strength was 3.80 MPa for the mix incorporating 10% RHA. Therefore, considering in view of practical implication according to the experimental results and waste exploitation, the performances of green mortar and concrete produced with POBC as an aggregate replacement can be improved by the optimum incorporation of binary mixtures of up to 10% CB, 30% RHA, and ternary mixtures of up to 5-10% CB with 10-20% RHA.

**Keywords**: Palm oil boiler clinker, Calcium bentonite, Rice husk ash, Compressive strength, Acid and sulfate durability, Chloride ion penetrability

## **Acknowledgements**

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This research would not has been possible without the support of Mr. Sombat Na-Bumroong, a friend of my father, who helped in exploring, collecting and delivering the palm oil boiler clinker from Surat Thani province.

I gratefully acknowledge the kind assistances that I received from friends, seniors, juniors, lecturers, and staffs whom I met at Department of Mining and Materials Engineering, Prince of Songkla University during this study.

Lastly, I'm deeply indebted to my parents and sister for send me their best wishes and unwavering supports. I also extremely grateful to my grandmothers, aunties, and cousins for providing me infinite helps and generosities.

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## **LIST OF ABBREVIATIONS**



## **LIST OF PUBLICATIONS**

- 1. Kueaket, K., & Tonnayopas, D. (2018). Enhanced properties of palm oil boiler clinker concrete with Sang Yod rice husk ash. *Proceeding of 7th International Conference on Applied Sciences and Engineering (ICASEA, 2018)*, Kota Kinabalu, Sabah, Malaysia, 29-38.
- 2. Kueaket, K., & Tonnayopas, D. (2019). Palm oil boiler clinker properties used as a green aggregate for construction. *Proceeding of the 13th International Conference on Mining, Materials, and Petroleum Engineering (CMMP 2019)*, Krabi, Thailand, 86- 91.
- 3. Kueaket, K., & Tonnayopas, D. (2021). Compressive strength and durability performance of mortar containing palm oil boiler clinker aggregate, rice husk ash, and calcium bentonite. *Journal of Applied Engineering Science*, 19(1), 193-203. doi: 10.5937/jaes0-27580
- 4. Kueaket, K., & Tonnayopas, D. (2021). Sustainable mortar and concrete made from palm oil boiler clinker aggregate comprising rice husk ash and calcium bentonite: Compressive strength and durability assessment. *Journal of Applied Science*, 20(1), 39-55. doi: 10.14416/j.appsci.2021.01.004



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#### NOTIFICATION OF PAPER ACCEPTANCE

Dear Ms. Kamolchanok Kueaket,

On behalf of the 7th ICASEA Committee, we are pleased to inform you that your submitted full paper (7th ICASEA: 043-025) entitled "ENHANCED PROPERTIES OF PALM OIL BOILER CLINKER CONCRETE WITH SANG YOD RICE HUSK ASH", has been ACCEPTED for the conference. Congratulation!

You may wish to update you Presenter Name. Simply Login > My Submission > Paper Submission > Click on "EDIT".

Please be informed that Conference Fee shall be paid by now. (Login > My Payment). Upload your proof of payment in the system after the payment is made.

As a reminder, the 7th International Conference on Applied Sciences and Engineering Application will be held on 8 & 9 December 2018 at TH Hotel, Kota Kinabalu, Sabah, Malaysia (Tabung Haji Hotel, Kota Kinabalu, Sabah). We look forward to seeing you at the conference.

#### Please be reminded that the Conference Fee shall be paid 2 weeks before the conference.

Again, thank you very much for your submission.

Secretariat 7th ICASEA Tel: +6010 342 3095 (Nadia) Fax: Email: icasea7@gmail.com Website: https://submit.confbay.com/conf/icasea7

## **CMMP2019 Acceptance Letter**



To: Kamolchanok Kueaket

Dear Kamolchanok Kueaket,

Thank you so much for your submission to the CMMP2019. We are pleased to inform you that your manuscript in the title of "PALM OIL BOILER CLINKER PROPERTIES USED AS A GREEN AGGREGATE FOR CONSTRUCTION" has been accepted for oral presentation.

The schedule of presentations will be informed later. We are looking forward to see you in 13-14 June 2019, Aonang Villa Resort, Krabi, Thailand.

However, the grammar and template of CMMP2019 should be checked again, and if reviewers have some recommended to your manuscript (details below of this email, or login to your EasyChair account), please correct it, and update before 25 April 2019.

Finally, the conference fee for early registration has been extended until 25 April 2019, please check the information for bank transfer and can make registration online at our website: http://cmmp2019.eng.psu.ac.th/

Best regards, CMMP2019 program chair

## [JAES][ID 27580] Editor Decision



SCIndeks Asistent <ceoncees@gmail.com> 26/10/2563 1:05

To: KAMOLCHANOK KUEAKET (กมลชนก เกื้อเกต); Danupon Tonnayopas

#### Dear Kamolchanok Kueaket, Danupon Tonnayopas,

It is my pleasure to inform you that your paper entitled COMPRESSIVE STRENGTH AND DURABILITY PERFORMANCE OF MORTAR<br>CONTAINING PALM OIL BOILER CLINKER AGGREGATE, RICE HUSK ASH, AND CALCIUM BENTONITE is accepted for publicat Journal of Applied Engineering Science.

With kindly regards,

Prof. Dr Gradimir Danon Editor in Chief gdanon@iipp.rs

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Ovaj mejl je poslat sa sistemskog naloga. Ako želite da odgovorite na njega, molimo Vas da koristite sledeću adresu e-pošte: This e-mail is sent from system account. To reply, please use the following e-mail address: "Prof. Dr Gradimir Danon" gdanon@iipp.rs

 $\rightarrow$ 

### [JASCI] Editor Decision



Asst.Prof.Dr.Nonchanutt Chudpooti via Thai Journals Online (ThaiJO) <admin@tci-thaijo.org> 22/3/2564 18:47

To: Kamolchanok Kueaket; Assoc. Prof. Dr. Danupon Tonnayopas

Save all attachments



Dear Kamolchanok Kueaket, Assoc. Prof. Dr. Danupon Tonnayopas,

We have reached a decision regarding your submission to The Journal of Applied Science, "Sustainable mortar and concrete made from palm oil boiler clinker aggregate comprising rice husk ash and calcium bentonite: Compressive strength and durability assessment".

Our decision: Submission accepted

Please fill the copyright form in the attachment and submit the form to ThaiJo system at your earliest convenience.

Best Regards, Asst.Prof.Dr.Nonchanutt Chudpooti nonchanutt.c@sci.kmutnb.ac.th

**Editorial Board** The Journal of Applied Science Faculty of Applied Science, King Mongkut's University of Technology North Bangkok  $\leftrightarrow$ 

## **Introduction**

Construction materials such as coarse aggregate, sand, and cement are the most important resources for public utility: road, dam, railway, airport, condominium, and other infrastructures that were built for facility and convenience. In Thailand, construction aggregate reserve was estimated at approximately 8,010 million tons from 318 quarries (Innovation in Raw Materials and Primary Industries Division, 2020). Specifically, limestone and shale reserves, which are the fundamental ingredients for cement production, was estimated at 2,245 million tons (Department of Mineral Resources, 2007). It can be seen that the reserve of nonrenewable construction materials was against the demand of aggregate for expanded construction sectors. According to Thailand rural roads standard.231-2562 (2019), 1  $\textsf{m}^{3}$  of lean concrete (1:3:5) contained 240 kg cement, 728 kg sand, and 1,218 kg coarse aggregate. Assumed that, the concrete demand is 50,000,000  $m<sup>3</sup>$  each year. Then, 61 million tons of coarse aggregate are consumed, annually. It could be predicted that the aggregate reserved will be completely depleted in AD 2151 (131 years afterwards).

Many researches introduced the sustainable constructions by substitute natural construction materials with industrial and agricultural wastes, recycle aggregates, construction materials with industrial and agricultural wastes i.e. oil palm shell (Ong et al., 2018), palm oil clinker (Huda et al., 2018), recycle aggregates from concrete and construction waste demolitions (Siddique et al., 2020), coal bottom ash, copper, and steel slag (Martínez-Lage et al., 2020) to alleviate the nonrenewable construction materials consumption. Green mortar has been produced by using local wastes for mitigate the environmental problems, land fill used, and disposal expenses (Yague et al., 2021).The green mortar, which contained 100% fly ash as a cement replacement (M-type or geopolymer mortar), also helped reducing carbon dioxide emission by 41% (Alqahtani et al., 2021).

In the southern part of Thailand, palm tree are the important economic plants that covered 7,812.82 square kilometers. Moreover, it was produced by 14,784,987 tons in 2020 (Office of Agricultural Economics, 2020) and fed to 70 local oil palm plants (Department of industrial works, 2020). Additionally, in Thailand, rice has been planted for 97,600 square kilometers, provided rice product for approximately 25,500,000 tons (Thai rice exporters association, 2020), and fed to 38,619 local rice mills (Office of agricultural economics, 2014). Thus, it is apparent that the large amount of agricultural and industrial wastes were generated unavoidably.

However, there is no official reports on quantity of wastes that were produced by rice mills and oil palm plants in Thailand. According to Suan et al. (2017) the palm oil wastes in Thailand (palm oil fuel ash (POFA) and palm oil boiler clinker (POBC)) were mentioned that they had a negative impact on environment and caused the disposal area problems. Those wastes are obtained from the burning of shell, bunch, kernel, and fiber of palm fruit.

Rice husk that obtained from rice mills almost utilized as a biomass fuel. Then, the rice husk ash (RHA) was provided after completed burning of rice husk. Chindaprasirt et al. (2008), Tonnayopas & Jitnukul (2013), and Raheem & Kareem (2017) revealed that RHA can be used as partial cement replacement and addressed as pozzolanic materials or supplementary cementitious materials (SCMs) due to the high percentage of amorphous silica component.

Calcium bentonite (CB) is clay mineral, which contained mostly  $SiO<sub>2</sub>$  and  $Al<sub>2</sub>O<sub>3</sub>$ (Velde, 1992). Few researches were conducted by using CB as a cement replacement to reduce the cost of construction (Mirza et al., 2009). Additionally, CB has an interesting properties: high plasticity and lubricity, low permeability and low compressibility (Memon et al., 2012). However, CB was reported that it provided low strength activity index when compared to reference cement mix (Achyutha Kumar Reddy & Ranga Rao, 2019; Al-Hammood et al., 2021; Mesboua et al., 2018).

Thus, in this study was employed the local waste by-products, i.e. POBC as 100% natural aggregate replacement, RHA and CB as partial substitution of ordinary Portland cement (OPC). Likewise, this research aims to impel and develop the green mortar and concrete for nonstructural application and promote waste utilization in local community: local oil palm plants and rice mills, as well as depleting the natural construction material consumption. The comprising between POBC aggregate, RHA, and CB will be studied in compressive

strength, durability properties, and microstructure to characterize the optimum proportion to produce the efficient performance green mortar and concrete for sustainable construction.

### **Objectives**

1. To investigate physical and chemical properties of palm oil boiler clinker (POBC) as coarse and fine aggregate, rice husk ash (RHA), and calcium bentonite (CB).

2. To investigate on physico-mechanical of green mortar containing POBC with RHA and CB as secondary cementitious materials (SCMs).

3. To evaluate on durability properties of green mortar and concrete.

## **Scope of study**

1. The properties of POBC coarse and fine aggregates were carried out including water absorption, specific gravity, unit weight, aggregate crushing value, Los Angles abrasion, and organic impurity.

2. The chemical composition and mineral phase compositions of POBC, RHA, and CB were confirmed by X-ray fluorescence spectrometry (XRF) and X-ray diffraction technique (XRD). In addition, the morphologies of POBC, RHA, and CB were observed under scanning electron microscope (SEM).

3. This study employed POBC as a fine aggregates, RHA and CB as SCMs in partial replacement by weight of OPC, type I at proportion of 10%, 20%, and 30%. All of binary and ternary mixtures will be blended with constant water to binder ratio (w/b) of 0.48 and cured in saturated-lime water at ambient temperature ( $29 \pm 3$  °C, RH=80-90%) for 7, 28, and 56 days.

4. Fresh properties of pastes and mortars: The fresh pastes of different binary and ternary mix designations were examined heat of hydration and setting time. Additionally, percentage flow and fresh density of fresh mortars were carried out.

5. Hardened properties of mortars: physical properties (water absorption and apparent porosity), compressive strength, strength activity index (SAI).

6. Durability properties of mortars and concretes blended with RHA and CB were determined sulfuric acid resistance, sodium sulfate resistance, and rapid chloride permeability test.

7. The microstructures and mineral phases of green mortars were investigated via scanning electron microscope (SEM) and X-ray diffraction technique (XRD).

## **Benefits**

1. Obtaining the guideline in sustainable environmental friendly solution for reducing wastes and promote a self-reliance in construction materials production by using local agricultural and industrial wastes for replacing the nonrenewable construction materials.

2. Further development and production of green mortar and concrete that have good performance in strength and durability.

### **Palm Oil Boiler Clinker (POBC)**

The industrial wastes generated from the palm oil mill are divided into two types i.e. fresh waste and biomass fuel waste. The fresh wastes include oil palm shell, empty fruit, and mesocarp fiber which are obtained after the crude palm oil extraction process. Furthermore, these fresh wastes, especially, the oil palm shell and mesocarp fiber were also used as a biomass fuel to generate the electricity for boiler and the palm oil mill. Then, the palm oil fuel ash (POFA) and the palm oil boiler clinker (POBC) were produced in the aftermath of the biomass fuel combustion. The range of the combustion temperature inside the boiler found to be varied from 500 to 1,200 °C (Hamada et al., 2020; Karim et al., 2018; Kueaket & Tonnayopas, 2018). The POBC's properties and characteristics can be varied depending on the temperature condition of the combustion (Kanadasan et al., 2015; Shakir et al., 2019).

### 1. Characteristics of POBC

### 1.1 Chemical compositions

According to the number of literature reports, the chemical compositions of POBC mainly consisted of 55 to 60%  $\mathrm{SiO}_2$ , 10 to 17% K<sub>2</sub>O, 4 to 8% CaO, and 2 to 5% MgO as tabulated in Table 1. Besides, the dissimilar chemical compounds of POBC can be found owing to the difference in the boiler operating condition: pressure and temperature, geological setting, and soil characteristics of oil palm cultivating area (Kanadasan et al., 2015; Shakir et al., 2019).

Chemical	Ahmmad et al.	Karim et al.	Nayaka et al.	Ismail et al.
composition (%)	(2017)	(2017)	(2019)	(2020)
SiO <sub>2</sub>	60.00	62.78	60.29	55.39
K <sub>2</sub> O	12.00	10.54		17.7
CaO	8.00	6.89	4.71	7.05
$P_2O_5$	5.00	۰		3.97
MgO	5.00	3.52		$\overline{2}$
Fe <sub>2</sub> O <sub>3</sub>	4.00	6.49	5.83	10.81
$\text{Al}_2\text{O}_3$	4.00	3.41	3.83	2.18
SO <sub>3</sub>		0.08	0.2	0.19
Others	2.00			0.28
Loss on ignition		3.67	2.01	0.02

Table 1 Comparison in chemical properties of POBC aggregates

## 1.2 Physical properties of POBC aggregate

The POBC was widely studied as a substitution of cement, sand, and aggregates for mortar and concrete productions, thus the physical properties of crushed POBC coarse and fine aggregates, included specific gravity, density, fineness modulus, and 24-h water absorption, had been reviewed as shown in Table 2 and Table 3. The POBC have been found in dark grey to greenish gray in color with irregular shape (Kanadasan et al., 2015; Kanadasan et al., 2018). The ranges of specific gravity of POBC coarse and fine aggregate were between 1.68 to 1.79 and 1.51 to 2.12, respectively. Accordingly, the density of POBC fine aggregate was in the range of 823 to 1,419 kg/m<sup>3</sup>. .

Specific gravity	Density $(kg/m^3)$	<b>Fineness</b> modulus	24-h Water absorption $(\% )$	References
1.78	823	6.23	5.7	Hamada et al. (2019)
1.75	568		5.67	Muthusamy et al. (2019)
1.68	813		5.56	Kabir et al. (2017)
1.9	1,419	4.99	4.23	Chai et al. (2017)
1.73			$3 + 2$	Abuhata et al. (2016)

Table 2 Physical properties of POBC coarse aggregate

Table 3 Physical properties of POBC fine aggregate

Specific gravity		<b>Fineness</b> modulus	24-h Water	
	Density		absorption	References
	$\left(\frac{\text{kg}}{\text{m}^3}\right)$		(%)	
1.51	945	5.89	5.50	Nayaka et al. (2019)
1.7	۰	3.16	19.9	Ong et al. (2018)
1.97-2.11		2.71	10±5	Kanadasan et al. (2015; 2018)
2.12	1,025	3.12	3.6	Ahmmad et al. (2017)

## **Utilization of POBC aggregate**

### 1. Use as a coarse aggregate replacement

Chai et al. (2017) stated that up to 50% of POBC could be used as coarse aggregate substitution for producing semi-lightweight high strength concrete. Utilization of POBC as a palm oil shell (POS) replacement could develop more UPV and compressive strength by 9% and 43% higher than that of the POS concrete due to the improvement of interlocking bonding between POBC particle and cement (Ahmmad et al., 2016). However, an increased in POBC as a coarse aggregate replacement up to 100% reduced the workability and 28-day compressive strength by 50% and 42% compared to that of control concrete (Abutaha et al., 2016). Hamada et al. (2019) recommended that the concrete containing 100% of POBC coarse aggregate had the higher water absorption than that of the control mix by 54% due to the irregular shape and porous of POBC particle, thus the mineral admixture need to be introduced for improving their durability properties. Moreover, Muthusamy et al. (2019) studied the suitable curing condition for POBC concrete and suggested that the POBC concrete need to be cured in water due to it need sufficient water for hydration process and pozzolanic reaction, while the air-cured specimen showed lower strength and durability properties compared to that of water-cured specimen. The comparisons in mix designation, compressive strength, and oven-dry density of concrete containing POBC as a coarse aggregate were tabulated in Table 4.

Size of POBC (mm)	w/b ratio	Maximum replacement (%)	28-day Compressive strength (MPa)	Oven-dry density $(kg/m^3)$	References
>4.75	0.34	50	68	2,202	Chai et al. (2017)
4.75-12.50	0.35	100	62	1,971	Ahmmad et al. (2016)
$5 - 14$	0.53	100	33	2,074	Abutaha et al. (2016)
>4.75	0.35	100	41	2,020	Hamada et al. (2019)
>4.75	0.45	100 with 50% of POBC fine aggregate as a sand replacement	60	1,850	Muthusamy et al. (2019)

Table 4 Compressive strength and oven-dry density of concrete containing POBC as a coarse aggregate with different mix designations.

## 2. Use as a fine aggregate replacement

The POBC was used to replace fine aggregate in 100% by weight and it provided the lower density mortar than that of the control mix by 11.3%. In addition, the porous content in POBC caused the strength reduction and weak interfacial transition zone (Kanadasan et al., 2018)**.** Meanwhile, some research pointed out that the rougher texture of POBC could improve the bonding connectivity of the interfacial transition zone (Lucas et al., 2016). The porosities of POBC was in the range of lesser than 10 microns to greater than 200 microns, hence an increase in POBC fine aggregate increased more water absorption and sorptivity. Likewise, the sorptivity had a good correlation to the thermal conductivity and thermal diffusivity (Asadi et al., 2021). Darvish et al. (2020) studied the geopolymer mortar comprising POBC as a whole sand replacement with 10wt.% fly ash, and found that an increase in molarity of NaOH concentration and fly ash contents increased the density of POBC geopolymer mortar. Kanadasan et al. (2015) compared a physical and mechanical properties of POBC mortar by using POBC from different states in Malaysia. They found that the strength properties was directly related to the characteristics of the POBC and the cost of construction could be reduced by 17% when replaced natural sand with the POBC fine aggregate. The comparisons in mix designation, compressive strength, and hardened density of concrete containing POBC as a fine aggregate were tabulated in Table 5.

Table 5 Compressive strength and hardened density of mortar containing POBC as a fine aggregate with different mix designations

Size of POBC (mm)	w/b ratio	Maximum replacement (%)	28-day Compressive strength (MPa)	Hardened density $(kg/m^3)$	References
$0.2 - 4$	0.30	100	65	2,015	Kanadasan et al. (2018)
4.75	0.5	100	33	1,890	Asadi et al. (2021)
4.75		100	20	1,710	Darvish et al. (2020)
4.75	0.28	100	80-100	1,950-2,260	Kanadasan et al. (2015)

#### **Calcium bentonite (CB)**

CB is a natural pozzolan and absorbent. It is impurity clay, which mostly composed of montmorillonite. The chemical compositions of some investigated CB are shown in Table 6. It can be seen that the  $SiO_2 + Al_2O_3 + Fe_2O_3$  contents of CB ranged between 75.34 to 86.36%. Specifically, the  $Al_2O_3$  contents ranged from 15.00% to 20.19%, where the  $Al_2O_3$ content of zeolite and metakaolin was 13.5% and 35.14% (Chen et al., 2018; Tafraoui et al., 2016). However, the composition of CB can be varied depending on geological setting, weathering process, quantity of Ca-montmorillonite and others minor minerals (Al-Hammood et al., 2021).

Chemical	Mirza et al.	Memon et al.	Ahmad et al.	Liu et al.
composition (%)	(2009)	(2011)	(2011)	(2020)
SiO <sub>2</sub>	49.44	54.55	65.00	64.32
K <sub>2</sub> O	0.69	3.92	0.27	1.74
CaO	7.45	7.28	2.66	7.13
$P_2O_5$		1.11		
MgO	1.61	4.20	6.5	3.68
Na <sub>2</sub> O	0.87	1.27	0.12	0.31
Fe <sub>2</sub> O <sub>3</sub>	6.20	8.60	3.00	3.80
$\text{Al}_2\text{O}_3$	19.7	20.19	15.00	18.24
SO <sub>3</sub>	-	-	$\overline{\phantom{a}}$	0.03
Loss on ignition (%)	13.74	5.46	6	

Table 6 Comparison in chemical properties of CB

The CB was determined by the major mineral phase composition of smectite clay group in them, which is calcium montmorillonite. The mineral structure of CB is similar to the sandwich, revealed by Man et al. (2019), as illustrated in Figure 1. It contained two tetrahedron layers and one octahedral layer. As a result, this structure causes the exchange ion capacity and water absorption ability of CB.



Figure 1 Schematic illustration of bentonite structure (Man et al., 2019).

## **Utilization of raw calcium bentonite as a supplementary cementitious materials (SCMs)**

Cost-reduction and environmental-friendly are the main purpose of using CB as a cement replacement. Thus, utilization of CB had been studied. CB was introduced as a lowcost pozzolanic material according to the study of Mirza et al. (2009), and the recommended CB ratio as a cement substitution was up to 20wt.% to minimize the compressive strength reduction (the 28-day compressive strength was lower than that of control specimen by 18%). Then, Memon et al. (2012) replaced up to 21wt.% of cement by CB and found that an increase in amount of CB decreased density and water absorption of concrete, while showed no significant effect to compressive strength. Ahmad et al. (2011) stated that an increase in amount of CB decreased prolonged compressive strength, which was confirmed by utilization of CB as a substitution of cement by up to 50wt.%. However, mortar containing 30wt.% of CB maintained durability against 5% sodium sulfate attack as compared to mortar without adding CB. Moreover, raw CB could be replaced cement slag by 25wt.% with w/b of 0.445 (2,493 kg of cement) or cement:sand:water ratio of 1:0.9:5. This mixture developed the 28 day compressive strength by 15% with respect to that of control mix, while this mixture with w/b of 0.485 effectively reduced the weight loss due to 5-cycle of sulfuric acid attack (Lee et al., 2021). Additionally, using 5-15% of CB could improve durability properties of eco-friendly concrete due to: (i) decrease water absorption and permeability by pore filling effect, (ii) reduce compressive strength loss after exposed toacid and sulfate attack (Rehman et al., 2020).

## **Rice husk ash (RHA)**

Thailand produced 25.5 million tons of rice in 2020 (*Thai Rice Exporters Association*, 2020). According to Sujivorakul et al. (2011), the rice husk has been estimated as 20wt.% of the total rice production. Then, after the rice husk was taken to use as a biomass fuel, the RHA was estimated as 4wt.% of the total rice production (At incinerated temperature of approximately 800-900 ºC). As a result, the RHA could be estimated as 1.02 million tons in 2020. The chemical composition of RHA contained approximately 80-95% of amorphous  $SiO<sub>2</sub>$  depended on the incinerating temperature and burning condition (Fapohunda et al., 2017).

			Burning condition	
References	$SiO2$ (wt.%)	<b>RHA</b> features	temperature	Duration
			$(^{\circ}C)$	(min)
Joshaghani &	87.10	Mostly amorphous RHA	700	60
Moeini (2018)		with density of 2.09 g/cm <sup>2</sup>		
		Mostly amorphous RHA		
Kannan & Ganesan	87.89	with density of 0.51 g/cm <sup>2</sup>	650	60
(2016)		and specific surface area		
		of 943 $m^2$ /kg		
Bheel et al. (2020)	86.94		600-700	300
	93.47	Mostly amorphous RHA	400-600	360-480
Isberto et al. (2021)		with pinkish-white color		
		Black RHA, partly	Approx.	
Sharma & Kumar	91.90	amorphous, contained	600-1,200	
(2021)		some crystalline silica	(open-air	
			burning)	
	96.11	Amorphous RHA with	700	1,440
Khan et al. (2020)		specific gravity of 2.83 g/cm <sup>3</sup>		

Table 7 Effect of burning condition to  $SiO<sub>2</sub>$  content and features of RHA

#### **Utilization of RHA as a supplementary cementitious materials (SCMs)**

RHA consisted of high amorphous silica content, which could reacted with  $Ca(OH)_2$ to form C-S-H phase as recognize as pozzolanic reaction and improve compressive strength and durability (Kumar et al., 2016; Suriyachoto & Tonnayopas, 2013). Tonnayopas & Jitnukul (2013) utilized local RHA (Sang Yod species) that was provided in the rice mill at Phatthalung province, Thailand. The RHA was gray in color with  $SiO<sub>2</sub>$  content of 66.13%, which was lower than those of other studies as shown in Table 7.This may be due to open-air burning condition and high residual unburned carbon. As a result, the maximum ratio of RHA replacement of cement was 10wt.% with w/b of 0.40, that could provide the compressive strength of 28-day concrete of 55 MPa. Accordingly, the maximum of 10wt.% of RHA was recommended to replaced cement for production coal bottom ash aggregate concrete with more durability and strength (Bheel et al., 2020). Moreover, the RHA that was burned in appropriate temperature of 700ºC for 60 minutes showed the high amorphousness and low unburned carbon.This RHA was replaced cement by weight in ranges of 10-30%, the mortar contained 20wt.% of RHA showed the highest compressive strength of 72 MPa, while an increase in RHA decreased the water absorption, chloride penetrability and weight loss due to acid attack. This was attributed to the pozzolanic activity of amorphous  $SiO<sub>2</sub>$  of RHA that reacted with portlandite (CaOH<sub>2</sub>) to produce secondary C-S-H by filled up the pore and lessened pore connectivity (Joshagani & Moeini, 2018). Alex et al. (2016) examined that RHA was derived by 5wt.% of rice husk, the RHA that was prepared for prolonged grinding time showed a lesser particle size with greater specific surface area, bulk density, and pozzolanic activity. Venkatanarayanan & Rangaraju (2014) investigated sulfate resistance properties of mortar containing low-carbon rice husk ash with w/b of 0.40, 0.48, and 0.57. An increase in RHA (up to 15%) with w/b ratio of 0.48 could lessen the 360-day expansion due to 5% sodium sulfate attack by 78% compared to that of control specimen. It could be explained by the pozzolanic activity of RHA improved impermeability matrix and prevented an entering of sulfate ion. Furthermore, the factors that effected the pozzolanicity of RHA were studied by many researchers, which can be concluded as follows (Fapohunda et al., 2017): (i) amorphousness of SiO $_2$ , (ii) specific surface area, (iii) size of RHA particles (fineness), and (iv) carbon contents. Additionally, the factors that effected strength and durability properties of concrete or mortar containing RHA as SCMs can be concluded as follows(Sandhu & Siddique, 2017): (i) ratio of cement substitution, (ii) curing time, (iii) w/b ratio, and (iv) mix design.

### **Effect of CB and RHA as SCMs on microstructure of hydration products under SEM analysis**

Rong et al. (2019) found that ultra-high performance cement-based composite containing the optimum 10-15% of RHA showed the improvement in mechanical properties. It can be seen in Figure 2, the microstructure of 7-day and 28-day's matrix of the mix containing 15% RHA composed of densely flocs-like C-S-H due to the pozzolanic reaction and some hydration products: portlandite (CaOH $_2$ ) and monosulfate (AFm). Yu et al. (1999) studied the pozzolanic reaction between SiO $_2$  from RHA and CaOH $_2$ . It was confirmed that C-S-H gel was formed in foil-like morphology in 28-day cement paste containing 10% RHA, as depicted in Figure 3. This finding recommended that incorporation of RHA in concrete could lessen the portlandite and increase durability and strength of the specimen due to the formation of C-S-H by this reaction. The fibrous-like C-S-H was clearly observed in microstructure of 4-h autoclaved aerated concrete containing RHA and aluminum powder (mixture containing: 45% OPC, 5% CaO, 45% RHA, 5% sand, and 0.5% aluminum powder) (Kunchariyakun et al., 2015), as presented in Figure 4



Figure 2 SEM images of ultra-high performance cement-based composite containing 15% RHA at the age of 7 days (A) and 28 days (B) (Rong et al., 2019)


Figure 3 SEM image of floc-like C-S-H found in paste containing 10% RHA at the age of 28 days (Yu et al., 1999)



Figure 4 SEM image of autoclaved aerated concrete containing RHA and aluminum powder (Kunchariyakun et al., 2015)

The microstructure of 28-day cement mortar blended with CB is shown in Figure 5. It can be seen that few flocs-like C-S-H was observed, however, the CB particle played the important role in filled up the large pores in matrix and improve the durability of mortar by filler effect (Liu et al., 2020). Lee et al. (2021) investigated slag mortars containing 0.25% and 0.50% CB. It is apparent that an increase in CB contents enlarged the voids in matrix. However, the C-S-H and ettringite (Aft) was observed in both mixes, as shown in Figure 6. This could be attributed to the layer crystal structure of CB and the absorption capacity, which absorbed more water and hindering the pozzolanic activity.



Figure 5 SEM image and EDS analysis of 28-day cement mortar blended with CB (Liu et al., 2020)



Figure 6 SEM image of slag mortars incorporating 0.25% CB (A) and 0.50% CB (B) (Lee et al., 2021)

# **RESULTS AND DISCUSSIONS**

# **Properties of POBC, RHA, and CB**

### 1. The physical properties of POBC aggregate

The large lumps of POBC as received from the palm oil mill are shown in Figure 7A. Each lump was approximately 10 to 25 cm in size. The POBC was light green to gray in color disseminated with some dark brown to black spots, irregular in shape, very angular, and scentless. The POBC coarse aggregate ( POBCCA) and fine aggregate (POBCFA) obtained after crushing process are presented in Figure 7B-C.



Figure 7 Large lumps of POBC (A), POBCCA (B), and POBCFA (C)

The grading requirement of fine and coarse aggregate was determined conforming to ASTM C33 (2018) . The grading analysis and physical properties of POBC aggregate are presented in Figure 8 and Table 8. The mean particle size  $(d_{50})$  of POBCCA and POBCFA was 10 mm and 0.55 mm, respectively. Moreover, the uniform grading analysis curve of POBC aggregate showed medium-graded and poorly-graded for the POBCCA and POBCFA, respectively. The fineness modulus of POBCFA also indicated that it was classified into the same size of coarse sand (0.5 to 1 mm) according to Wentworth scale of rock particle sizes. The Los Angeles abrasion and aggregate impact value of POBCCA represented that the POBC had low resistance to abrasion and crushing. Thus, the POBCCA was not recommended for using in road surfacing, railway ballast, and structural construction purpose.The POBC aggregate contained none of organic impurity, as can be seen in Figure 9.



Figure 8 Grading analysis of POBC aggregates

Properties	<b>POBCCA</b>	<b>POBCFA</b>	
Water absorption (%)	1.48	3.15	
Specific gravity	2.20	2.40	
Unit weight $(kg/m^3)$	1,182	1,350	
Los Angeles abrasion value (%)	37.98		
Uniformity factor	0.24		
Aggregate impact value (%)	37.30		
Particle size distribution			
$d_{10}$ (mm)	4.00	0.35	
$d_{50}$ (mm)	10.00	0.55	
$d_{90}$ (mm)	15.71	1.86	
Fineness modulus	5.42	3.74	
Organic impurity	None		

Table 8 Physical properties of POBC aggregates



Figure 9 The organic impurity test of POBC aggregate as per ASTM C40

### 2. The grading analysis and physical properties of RHA and CB

The grading curves of RHA and CB were plotted and compared to that of the OPC, as shown in Figure 10. It is evident that the particle sizes of RHA and CB were finer than the OPC. The mean particle size  $(d_{50})$  of RHA and CB was 12.5 and 2.3 µm, which was lower than that of the OPC by 10% and 84%, respectively. The percentage retained on 45 µm of RHA and CB was 5% and 0.3%, respectively. According to ASTM C618 (2019), the fineness requirement of percentage retained on 45 µm was not exceeded than 34% for the pozzolanic materials class N, C, and F. Thus, the RHA and CB particle sizes were met the fineness requirement. Moreover, the true density (ASTM C604) values of RHA and CB were 2.39 and 2.47, which were lower than that of OPC by 22% and 20%, respectively, as listed in Table 9. Similarly, the OPC also had the highest specific gravity value (3.1) compared to RHA (2.4) and CB (2.5). The true density values conformed to the specific gravity values, as it represented the voids between particles which was directly proportional to the specific gravity of the OPC, RHA, and CB.



Figure 10 Grading analysis of OPC, RHA, and CB

Properties	<b>OPC</b>	<b>RHA</b>	CB.
Moisture content (%)	0.33	0.34	0.51
True density ( $g/cm3$ )	3.08	2.39	2.47
Specific gravity	3.10	2.40	2.50
Particle size distribution			
$d_{10}$ (mm)	1.9	2.3	0.6
$d_{50}$ (mm)	13.9	12.5	2.3
$d_{90}$ (mm)	32.7	38.6	14.2
Percentage retained on 45 µm	1.4	5	0.3

Table 9 Physical properties of OPC, RHA, and CB

# 3. The chemical compositions of POBC, RHA, and CB

The chemical compositions of POBC, RHA, and CB were analyzed via X-ray fluorescence (XRF) spectrometer, and tabulated in Table 10. It can be seen that the main constituents of POBC, RHA, and CB were  $SiO<sub>2</sub>$ . In addition, the inferior compositions of POBC were CaO and  $K_2O$ , while those of CB was  $Al_2O_3$ . Moreover, the composition of CB consisted of more CaO in the proportional of approximately 10% than NaO. Additionally, the XRD patterns of POBC, RHA, and CB were carried out via X- ray diffraction ( XRD) spectrometer, and mineral phase compositions are illustrated in Figure 11. The mineral

composition of POBC consisted of quartz (  $2\theta$  = 26.62° and 50.10°), tridymite (  $2\theta$  = 20.51° and 23.23°), crystobalite (  $2\theta$  =  $21.95^{\circ}$ ), whitlockite (  $2\theta$  =  $27.99^{\circ}$ , 31.26°, and 34.60°), and diopside (  $2\theta$  =  $29.85^{\circ}$ ,  $35.63^{\circ}$ , and  $54.79^{\circ}$ ). The polymorphs of SiO $_2$  found in XRD pattern also indicated that the temperature in the boiler was in the range of 870°C to 1,470°C, while the POBC was formed after the combustion process of palm oil waste (Di Febo et al., 2020). In addition, the broad hump in the background intensity between 2theta 20° to 30° implied the amorphous phases. The XRD pattern of RHA is presented in Figure 12. It can be clearly seen that RHA composed mainly of amorphous silica (broad peak) and some crystalline silica i. e. , quartz and crystobalite. The mineral phase compositions of CB are demonstrated in Figure 13. CB consisted of quartz (  $2\theta$  = 20.84° and 26.62°), montmorillonite ( $2\theta$  = 5.72°, 19.82°, 34.08° and 61.94°), kaolinite ( $2\theta$  = 12.36° and 24.87°), and gypsum  $(2\theta = 11.63^{\circ} \text{ and } 23.39^{\circ}).$ 

Oxides (wt.%)	<b>POBC</b>	<b>RHA</b>	CB
$\text{Al}_2\text{O}_3$	1.66	0.66	19.81
SiO <sub>2</sub>	65.23	93.83	56.62
Fe <sub>2</sub> O <sub>3</sub>	1.78	0.36	0.02
CaO	10.00	0.76	1.16
Na <sub>2</sub> O	0.13	0.07	0.18
SO <sub>3</sub>	0.04	0.21	0.69
MgO	5.37	0.36	0.55
K <sub>2</sub> O	9.56	1.77	0.96
$P_2O_5$	5.50	1.04	0.05
Loss on ignition	0.04	0.67	10.12

Table 10 Chemical compositions of POBC, RHA, and CB by semi-quantitative XRF analysis



Figure 11 XRD pattern and mineral phase compositions of POBC



Figure 12 XRD pattern and mineral phase compositions of RHA



Figure 13 XRD pattern and mineral phase compositions of CB

# **SEM morphological analysisof POBC, RHA, and CB**

The morphological analysis of POBC, RHA, and CB was obtained via scanning electron microscope (SEM), 20 kV. The SEM images of POBC, RHA, and CB are presented in Figure 14A- B. The POBC obtained after crushing to fine aggregate was very angular particle and low sphericity with irregular shape. An enlargement of POBC surface is depicted in Figure 14C. In addition, the POBC had a smooth surface consisted of a few micro porous and the agglomeration of silica component (a smooth rounded shape) , as can be seen in Figure 14D.

The RHA ( Sang Yod species) obtained after grinding process is shown in Figure 15A- B. The particle shape of RHA was sub-angular to sub- rounded and medium sphericity. It can be noticed that the RHA particle sizes were varied from coarse to very fine particle. An enlargement of RHA surface demonstrated the clustered feature of silica component, which was the principal composition of RHA, as depicted in Figure 15B.



Figure 14 Morphology of POBC aggregate (A-B) and characteristics of POBC surface's texture (C-D)

The morphology of CB is illustrated in Figure 15C-D. The compactness of the sheet-like structure of a small CB particle represented to the clay mineral characteristics. The clump of sheet-like CB particle was rounded in shape and high sphericity. Besides, the subangular particle of OPC is shown in Figure 15E. It can be seen that the OPC contained poorly graded coarser particle sizes than those of RHA and CB. The combination of OPC, RHA, and CB particles showed well graded characteristic and dense particle packing with the different particle sizes and shapes, as demonstrated in Figure 15F.



Figure 15 Morphology of RHA (A-B), CB (C-D), OPC (E), and ternary blends of OPC, RHA, and CB particles (F)

#### **Properties of paste incorporating CB and RHA**

#### **Setting time by Vicat needle test**

In this study, the test method for evaluate Vicat initial and final time of setting was adapted from ASTM C191 (2008). The Vicat initial setting time was carried out by measuring the elapsed time until the penetration depth of Vicat needle was 25 mm, while the Vicat final time of setting was determined as the Vicat needle failed to pierce the mold. The Vicat setting time of different mix designations are presented in Figure 16. It is evident that both of the Vicat initial and final setting time decreased with an increase in percentage of RHA replacement. For the ternary blends of RHA and CB, the same trend of Vicat initial and final setting time was found to be decreased with an increase in percentage of RHA replacement. Besides, the Vicat initial and final setting time of mixes containing 10% to 30% RHA ranged from 96 to 133 minutes and 240 to 285 minutes, which decreased up to 32% and 16% as up to 30% of RHA replacement, when compared to that of the control mix. These findings are in agreement with the previous studies on the effect of RHA as a cement replacement on setting time. According to Liu et al. (2020) and Venkatanarayanan & Rangaraju (2015) , the RHA particle absorbed more free water, which in turn reduced the water to binder ratio of the fresh paste and setting time. Meanwhile, an increase in CB contents slightly increased either the Vicat initial and final setting time, which were in the range of 199 to 211 minutes and 300 to 320 minutes, respectively. At up to 30% CB replacement, the Vicat initial and final setting time were 50% and 12% greater than that of the control. The previous studies also found that the setting time increased with increasing the CB contents, this is due to the packing effect of CB which could hindered and delayed the hydration process (Sha et al., 2018; Zhou et al., 2020).



Figure 16 Vicat initial and final setting time of different binary and ternary mix designations

#### **Temperature of hydration**

The temperature of hydration was measured during the setting time had been carried out, by using Fluke Digital Multimeters ( DMMs) . For all mix designations, the temperature of hydration was measured every 15 minutes until the paste hardened and the results are demonstrated in Figure 17. Generally, the heat of hydration is chiefly generated from tricalcium aluminate and free lime in cement contents, hence a decrease in cement contents by using pozzolans as a cement substitution decreases the hydration temperature due to a decrement of tricalcium aluminate and free lime (Gómez & Rojas, 2013) . The hydration temperature ranged from 28.4 to 31.2 °C for the mix designation containing RHA and CB, while that of control mix was 33.6 °C. Particularly, the maximum binary substitution of OPC by 30% of RHA, CB, or ternary blends of RHA and CB provided the lower hydration temperature than that of the control by 9 to 14%. Wei & Gencturk (2019) studied the hydration of blends containing bentonite and stated that the dilution effect by reducing the use of cement could decrease the heat of hydration due to lower cement take part in both of hydration reaction and tricalcium aluminate reaction.



Figure 17 Temperature of hydration of different binary and ternary mix designations of pastes

### **Fresh properties of POBC mortar**

## 1. Percentage of flow

The flowability test of fresh POBC mortars was performed in accordance with (2015). The percentages flow of different binary and ternary mix designations are presented in Figure 18. Generally, an increase in RHA and CB contents decreased the flowability of POBC mortar. For the binary mix designation of R10, R20, and R30, the percentages of flow were lower than that of control mix by 19%, 31%, and 38%, respectively. Additionally, the percentage flow of the binary mix containing up to 30% CB was lesser than the control mix by 20%. Subsequently, an increase in percentages of cement substitution by the ternary blends of RHA and CB decreased the flowability. However, the mix incorporating up to 10% RHA, 20% CB, and ternary mix designation of B5R5, B5R15, and B15R5 showed the acceptable percentages of flow within the range of 98% to 119%. Laidani et al. (2020) also revealed that the flowability of concrete containing CB decreased with an increase in CB contents due to the fineness and specific surface area of CB particle were greater than that of cement, therefore, the more water was required. Subsequently, the percentage of flow

gradually decreased with an increase in RHA replacement ratios for the ternary blends of RHA and CB (Figure 18). This finding is in accordance with those of previous studies. Siddika et al. (2020) pointed out that RHA particle absorbed water on its surface and stored water in its pore, an increase in RHA contents and fineness of RHA particle decreased free water, which in turn increased the viscosity of the fresh mix and decreased the flowability. As well as, the filler effect of RHA played the important role in reducing the segregation and flowability of fresh mix (Fapohunda et al., 2017). In addition, Sua-Iam et al. (2019) and Abbas et al. (2017) suggested that more superplasticizer dosages are required to restore flowability and achieve the targeted flow of 110±5% for the mixture incorporating RHA.



Figure 18 Percentage flow of different binary and ternary mix designations

### 2. Fresh density

The fresh density of POBC mortars with different mix designations was measured according to ASTM C138 (2017) and demonstrated in Figure 19. It can be seen that the fresh density decreased with an increase in RHA and CB contents. For the mix incorporating 10%, 20%, and 30% RHA, the fresh density was 3%, 6%, and 8% lower than that of control mix. Similarly, an increase in CB partial replacement of OPC from 10% to 30% reduced the fresh density by 2% to 5% compared to the control mix. The fresh density of the ternary blends of RHA and CB ranged 2% to 6% lower than that of the control mix. It is on account of the fact that the fresh density was the function of the true density and specific gravity. The true density and specific gravity values of OPC were greater than that of RHA and CB. Consequently, the control mix had the highest fresh density compared to the rest of mix designations. This result is consistent with the previous studies. Memon et al. (2012) revealed that the fresh density of mortar containing up to 21% CB was lower than that of without CB by 7%. Furthermore, an increase in the percentage of OPC substitution by RHA up to 20% decreased the fresh density of mortar by 2.5% compared to the control mix, due to the specific gravity of RHA (2.2) was lower than that of OPC (3.2) (Sua-iam et al., 2016).



Figure 19 Fresh densities of different binary and ternary mix designations

#### **Properties of POBC mortars**

### **Water absorption and apparent porosity**

The water absorption and apparent porosity were conducted conforming to ASTM C373-14a (2014) . The water absorption and apparent porosity results of POBC mortars with different binary and ternary mix designations at the ages of 7, 28, and 56 days, as presented in Figure 20. An increase in RHA contents increased the water absorption at the early curing age (7 days) , while, at the prolonged curing periods, the water absorption decreased with an increase in RHA contents. For instance, the water absorption of 56-day POBC mortars containing 10%, 20%, and 30% RHA was 21%, 28%, and 35% lesser than that of the control mix, respectively. Similar result was confirmed by Balraj et al. (2020) that the water absorption of 28-day concrete decreased with a reduction in particle size of RHA and an increase in RHA contents. The possible reason could be due to the reduction of permeable void by the pozzolanic effect of RHA, which took place at the later ages (up to 28 days of curing), whereas an increase in water absorption at the early age could be attributed to the absorption capacity of the porous RHA particle (Ganesan et al. , 2008) . For the mix containing CB, the water absorption decreased with prolonged curing ages and a reduction in CB content. For example, the minimum 56-day water absorption value was found in the mix containing 10% CB, which was lesser than that of the control mix by 13%, whereas the mortar containing up to 30% CB showed greater water absorption value than that of control mix by 13%.

Accordingly, the apparent porosity results were in same trend of the water absorption results, as presented in Figure 20. An increase in RHA contents and curing ages decreased the apparent porosity due to the filler effect of RHA and the secondary C- S-H formed in the pore of the mortar specimen. For example, the 56-day mortar containing 30% RHA showed the lowest apparent porosity (14%) , which was 33% lower than that of the control mix. Conversely, an increase in CB contents increased the apparent porosity of the mortars, while the substitution of OPC by 10% CB reduced the 56-day apparent porosity of mortar by 14% compared to the control mix. The possible reason could be due to the absorption capacity of more CB particles that absorbed more water and swelled in the fresh

mix, when the water released, the pore size of approximately 1 µm was produced (Hu et al., 2019). Thus, the optimum substitution of OPC by 10% CB was recommended based on this study result. Moreover, a decrease in porosity was observed in the mix containing the optimum amount of 10-15% CB as an OPC substitution, which was reported by Laidani et al. (2020).This finding is in line with Man et al. (2019) and Targan et al. (2002).



Figure 20 Water absorption and apparent porosity of different binary and ternary mix designations and curing ages

## **Compressive strength**

The compressive strength of POBC mortars with different mix designations at 7, 28, and 56 days was determined conforming to ASTM C109 (2016) . As can be clearly seen in Figure 21, the compressive strength of each mix designation increased with curing ages. The development of compressive strength for the mix incorporating RHA was observed at up to 28 days of curing period. The highest compressive strength was found in the binary mix designation of R20, which was 45 MPa and 53 MPa at the age of 28 and 56 days, respectively. However, at an early curing period of 7 days, an increase in RHA contents decreased the compressive strength. This is due to the pozzolanic effect, which is thereaction

between SiO<sub>2</sub> derived from RHA and Ca(OH)<sub>2</sub> from hydration process. This reaction was confirmed by Hu et al. (2020) that it took place at later age (up to 28 days) and produced calcium silicate hydrate (C-S-H), which resulted in strength development. Similar results was reported by Khan et al. (2018) . Nevertheless, an increase in CB contents decreased the development of compressive strength. The highest compressive strength of the mix incorporating CB was 41 MPa at the age of 56 days, which was found in the 10% CB supplemented mix. The reduction in compressive strength of that mix was only 11% compared to the control mix. For mix designation containing the ternary blends of RHA and CB, the highest compressive strength was observed in the mix designation B5R15, which was 39 MPa and 48 MPa, respectively, at the age of 28 and 56 days. The possible reason is CB contained 57%  $\,$  SiO $_2$  in comparison with 94%  $\,$  SiO $_2$  of RHA, thus the mix designations incorporating RHA and/or more RHA contents provided more compressive strength values than those of CB. Moreover, an addition of excessive CB could hinder the hydration and pozzolanic reaction. This finding is in line with Ahmad et al. (2011) and Mesboua et al. (2018).



Figure 21 Compressive strength of different binary and ternary mix designations and curing ages

### **Strength activity index (SAI)**

The SAI is the ratio between the compressive strength of mortar containing pozzolanic materials to the compressive strength of the control mix. The SAI was calculated and tested as per ASTM C311 (2018) and C618 (2019). According to ASTM C618 (2019), the minimum standard requirement of SAI was 75% of the control mix. It can be seen that the SAI increased with prolonged curing ages, as illustrated in Figure 22. For all of the mix incorporating RHA and the ternary blends of RHA and CB at the ages of 7, 28 and 56 days, the SAI values were met the standard requirement. Regarding to the compressive strength results, the same trend was observed in the SAI. The highest SAI was found to be 127% and 107%, respectively, for the mix designation of R20 and B5R15 at the age of 28 days, whereas, the mix incorporating up to 10% CB provided the highest SAI (83% and 91%) at the ages of 28 and 56 days. However, an increase in CB contents decreased the SAI, similar results were reported by Mesboua et al. (2018) and Mirza et al. (2009). Additionally, Abbas et al. (2017) concluded that substitution of OPC by up to 30% RHA with the curing age up to 28 days could be capably in strength development due to the 28-day SAI was exceeded than 75% of the control mix.



Figure 22 Strength activity index of different binary and ternary mix designations and curing ages

#### **Sulfuric acid attack**

The different mix designations of the POBC mortar specimens cured in water for 7, 28 and 56 days, were then exposed to 0.005 M (0.1% w/v, pH=2.5) sulfuric acid solution (H<sub>2</sub>SO<sub>4</sub>) for 6 weeks (42 days). The weight of each specimen was measured before and after exposure. The change in compressive strength was examined by comparison of the compressive strength at particular curing age of 7, 28, and 56 days before exposure with those of after exposure to sulfuric acid.

#### 1. Loss in weight

The percentage loss in weight of different mix designations is revealed in Figure 23. It is apparent that the percentage loss in weight gradually decreased with prolonged curing age before exposure. Additionally, an increase in RHA decreased the percentage loss in weight. The 56-day percentage loss in weight was 0.81%, 0.68%, and 0.47% for the binary mix designation of R10, R20, and R30, respectively, which was 47%, 56%, and 69% lower than that of the control mix. For the binary mix designation B10, B20, and B30, the 56-day percentage loss was 0.97%, 1.54%, and 1.57%, which was 37% lower than that of the control mix; 1%, and 3% greater than that of control mix, respectively. For the ternary mix designation B5R5, B5R15, B15R5, B10R20 and B20R10, the 56-day loss in weight was 1.14%, 0.86%, 1.47%, 0.90% and 1.52%, respectively. The reaction between cement paste and sulfuric acid solution caused the dissolution of calcium and solid phases ( Subashi De Silva et al. , 2021) . As a result, the POBC mortars exposed to sulfuric acid showed the deteriorating characteristics i.e. change in surface's color, broken rim, and loss in weight, as revealed in Figure 24. Similar result was also reported by Subashi De Silva et al. (2021) that an increase of up to 20 wt.% RHA replacement of OPC decreased the weight loss due to sulfuric acid attack by 25%. This could be due to the pozzolanic and filler effect of RHA reduced both pore-connectivity, permeability, and sorptivity by C- S-H forming in the micro-pore spaces. Thus, the weight loss was reduced by an incorporation of RHA as an OPC replacement.

### 2. Loss in compressive strength

The percentage loss in compressive strength of different mix designations is presented in Figure 25. The trend of percentage loss in compressive strength was in correspondence to those of loss in weight (Figure 23). The percentage loss in compressive strength decreased with age of specimens before exposure to sulfuric acid solution. The 56 day percentage loss in compressive strength for the binary mix designation of R10, R20, and R30 was 23%, 41%, and 68% lower than that of the control mix, respectively. For the mix designation, B10, the percentage loss in compressive strength was 11% lesser than that of the control mix. Conversely, the 56-day percentage loss in compressive strength for the binary mix designation of B20 and B30 was 12% and 23% greater than that of control mix. For the ternary mix designation of RHA and CB, the 56-day percentage loss in compressive strength was in the range of 4.07% to 4.93%, where the lowest percentage loss performed by in the ternary mix designation of B5R15; which was lower than that of the control mix by 18% . This result is in agreement with the research of Kumar & Prasad (2019) . The compressive strength loss could be due to the loosened surface layer of mortar and micro crack; which was induced by the formation of gypsum and ettringite via the reaction between sulfuric acid and hydration products (monosulfoaluminate, C-S-H or calcium hydroxide) (Sata et al., 2012).

Based on the sulfuric acid attack mechanism, the reaction between hydration products and sulfuric acid solution is revealed as follows (Monteny et al., 2000):

$$
Ca(OH)2 + H2SO4 \rightarrow CaSO4.2H2O
$$
\n(1)  
\n(Pysum)

$$
CaO.SiO2.2H2O+ H2SO4 \rightarrow CaSO4 + Si(OH)4 + H2O
$$
\n(C-S-H) (Gypsum)

$$
3CaO.AI2O3.12H2O + 3(CaSO4.2H2O) + 14H2O → 3CaO.AI2O3.3CaSO4.31H2O
$$
\n(C<sub>3</sub>A) (Gypsum) (E  
Effingite formed in pore solution)

As a result, the weight loss could be explained by the dissolution of calcium of cement's paste. The mortar's structure weakened by the dissolution of that solid phase and the forming of new salt's phase. Then, the compressive strength declined consecutively.



Figure 23 Percentage loss in weight of different binary and ternary mix designations and curing age after sulfuric acid attack



Figure 24 Visual observation of deteriorating characteristics of POBC mortars exposed to sulfuric acid solution for 42 days (from left to right: control mix, R10, R20, and R30)



Figure 25 Percentage loss in compressive strength of different binary and ternary mix designations and curing ages after sulfuric acid attack

# **Sodium sulfate attack**

1. Gain in weight

The POBC mortar specimens with different binary and ternary mix designations were cured in water for 7, 28 and 56 days, then were exposed to 0. 5 M (8% w/v, pH=6-8) sodium sulfate solution (Na<sub>2</sub>SO<sub>4</sub>) for 6 weeks (42 days). Each specimen was weighed before and after exposure. The change in compressive strength was performed by comparison of the compressive strength at particular curing age of 7, 28, and 56 days before exposure with those of after exposure to sodium sulfate solution for 42 days.

It is apparent that the weight of each different mixtures slightly increased after exposure to sodium sulfate solution. However, an increase in curing age before exposure and RHA contents decreased the gain in weight. The percentage gain in weight of different mix designations was demonstrated in Figure 26. The 56-day percentage gain in weight was 0.09%, 0.07%, 0.03%, 0.12%, 0.15%, and 0.16% for the binary mix designation of R10, R20, R30, B10, B20, and B30, respectively. Additionally, the 56-day percentage gain in weight of the ternary mix designation of B5R5, B5R15, B15R5, B10R20, and B20R10 was 0.12%, 0.09%, 0.13%, 0.10%, and 0.14%, respectively. The lowest 56-day percentage gain in weight was found in the mix designation of R30, which was 76% lesser than that of the control mix. Conversely, an increase in CB content up to 30% extended the 56-day percentage gain in weight to 23% compared to that of the control mix. Proportionately, the ternary mix designation between RHA and CB manifested lesser 56-day percentage gain in weight than the mix incorporating only CB. It could be attributed to ( i) the specimen was saturated by absorption of sodium sulfate solution (ii) CB was rich in alumina which could conduce more C- A- S-H and sulfate salts forming ( iii) the formation of sulfate salt on the surface of mortar's specimen, as depicted in Figure 27. This results are in agreement with Ma et al. (2014). Additionally, this result conformed to water absorption and apparent porosity results, where RHA had a filler effect to POBC mortar's matrix and reduced the penetrating ability of sulfate solution to the specimen.



Figure 26 Percentage gain in weight of different binary and ternary mix designations and curing ages after sodium sulfate attack



Figure 27 Visual observation of deteriorating characteristics of POBC mortars exposed to sodium sulfate solution for 42 days: sulfate salt precipitated on specimen's surface (A) and sulfate salt deposited as a plate inside cementitious material (B).

### 2. Loss in compressive strength

The degradation in compressive strengths after exposure to sodium sulfate solution for 42 days were monitored and represented as percentage loss in compressive strength, depicted in Figure 28. The result demonstrated the similar trend to the percentage gain in weight after exposure. In addition, an increase in curing age before exposure significantly declined the percentage loss in compressive strength. The 56-day percentage loss in compressive strength was 0.85%, 0.56%, and 0.32%, respectively, for the binary mix designation of R10, R20, and R30. For the binary mix designation of B10, B20, and B30, the 56-day percentage loss in compressive strength was 0.98%, 1.21%, and 1.32%, respectively. Consequently, the lesser values of 56-day percentage loss in compressive strength was observed in the ternary mix designation of B5R5, B5R15, B15R5, and B10R20. As compared to the control mix, an incorporating of up to 30% RHA, 10% CB, and the ternary mix of 5% CB and 15% RHA, provided the lower 56-day percentage loss in compressive strength than that of the control mix by 72%, 15%, and 27%, respectively. This result is in accordance with Hu et al. (2020). Based on the external sulfate attack mechanism that was proposed by Lv et al. (2020) , the reaction between hydration products and sodium sulfate solution is demonstrated as follows:

$$
Ca(OH)2 + Na2SO4 + 2H2O \rightarrow CaSO4.2H2O + NaOH
$$
 (Pysum) (Gypsum)

$$
CAH + Na2SO4 + 2H2O \rightarrow 3CaO.AI2O3.3CaSO4.31H2O + NaOH
$$
 (CAH or 3CaO.AI<sub>2</sub>O<sub>3</sub>.6H<sub>2</sub>O (Ettingite)  
from hydration reaction of C<sub>3</sub>A)

$$
CaSO4.2H2O + CAH \rightarrow 3CaO.AI2O3.3CaSO4.31H2O + Ca(OH)2
$$
 (Gypsum, as formed in equation 4) (Etringite)

The hydration products, i.e., porlandite and C-A-S-H reacted with the sulfate solution to form gypsum and ettringite. As a result, the formation of gypsum and ettringite deposited nearly the specimen's surface induced microstructural alteration, expansion, strength loss, and micro cracks (Santhanam et al., 2003).



Figure 28 Percentage loss in compressive strength of different binary and ternary mix designations and curing ages after sodium sulfate attack

#### **X-ray diffraction analysis**

The crystalline phase compositions of each mix were carried out using X- ray diffractometer at 2theta between  $4^{\circ}$  to 60° with step size of 0.026° and time $\cdot$ step<sup>-1</sup> 127.50 seconds, respectively. Table 11 demonstrates the abbreviations of mineral phases labeled on XRD spectra and their peak positions of different mixes in accordance with the ICDD database (International center for diffraction data).

The XRD patterns of the different mix designations (control, R30, B10, and B5R15) at the age of 56 days are illustrated in Figure 29. The main hydration's products were found in all mixes, which were ettringite  $(\, \text{Ca}_{6}\text{Al}_{2}(\, \text{SO}_{4})_{\,3}(\, \text{OH})_{\,12}(\, \text{H}_{2}\text{O})_{\,26})$ , portlandite ( Ca( OH) <sub>2</sub>, quartz (SiO<sub>2</sub>), dicalcium silicate (  $\text{Ca}_2\text{SiO}_4$ ), and calcium silicate hydrate (Ca<sub>1.5</sub>SiO<sub>3.5</sub>·<sub>x</sub>H<sub>2</sub>O, C-S-H), while calcium aluminum silicate hydrate (Ca<sub>2</sub>Al<sub>4</sub>Si<sub>14</sub>O<sub>36</sub>·14H<sub>2</sub>O, C-A- S-H) was found in the mix designation B10 at 2theta of 9.79° and 29.76°, respectively. However, C-S-H is an amorphous, which can be detected at low angle scattering (Hou et al., 2015), exhibited the broad and hump peak in the background intensity at 2theta of 29.36 $^{\circ}$ and 50.08°, respectively.

It is evident that the peak intensities of portlandite for the mix designation R30, B10, B5R15 were lower than that of the control mix. In addition, the mix designation R30 presented the highest C-S-H and  $C_2S$  peak intensities, followed by B5R15, B10, and control mix (Figure 29). This is indicated that RHA and CB could be effectively used as a pozzolan due to ( i) the lower peak intensity of portlandite and the greater peak intensity of C-S-H was observed in the mix incorporating RHA, (ii) the incorporating of CB also reduced the peak intensity of portlandite and promoted the peak of C- A- S-H, which could attributed to the more alumina composition of CB interacted with OPC (Fernandez et al. , 2016) . Moreover, the peak of ettringite and  $C_2S$  also indicated the primary and prolonged strength development (Jeong et al., 2016; Saghiri et al., 2017). The pozzolanic reaction could be promoted by the  $SiO<sub>2</sub>$  content of RHA consumed Ca(OH)<sub>2</sub> and generated more secondary C-S-H (Jamil et al., 2016).Furthermore, the incorporating of RHA in recycled aggregate concrete could promoted the 28-day compressive strength and durability against hydrochloric acid attack by pozzolanic effect that increased the C-S-H formation (Alnahhal et al., 2018).

Abbreviation	Mineral phase	Peak position at 2theta (degree)
E.	Ettringite	9.72°, 15.77°, 22.90°, and 46.66°
P	Portlandite	18.01°, 28.67°, 34.10°, and 50.81°
Q	Quartz	20.84°, 21.82°, 26.62°, and 39.43°
$C_2S$	Dicalcium silicate (larnite)	$32.02^{\circ}$
$C-S-H$	Calcium silicate hydrate	$29.36^{\circ}$ and $50.08^{\circ}$
$C-A-S-H$	Calcium aluminum silicate hydrate	$9.79^{\circ}$ and $29.93^{\circ}$

Table 11 The mineral phases and peak positions of XRD analysis



Figure 29 XRD patterns of different binary and ternary mix designations

#### **Microstructural characterization**

The SEM images of POBC specimens cured in water with different ages and mix designations are demonstrated in Figure 30. Generally, it can be seen that the hydration products of different mix designations formed in different sizes and shapes i.e. portlandite (P), ettringite (E), calcium silicate hydrate (C-S-H) in flocs-like and fibrous-like forms. The microstructural analysis was carried out based on the XRD results and crystallographic properties i.e. shapes, habits, crystal forms, crystal systems, and cleavage. Portlandite (Ca(OH)<sub>2</sub>) is a hexagonal-like plate, which is classified into hexagonal crystal system and exhibited one perfect cleavage. It was formed by the hydration reaction and can generate the secondary C- S-H with the pozzolanic materials (Neville & Brooks, 2010) . The negative effect of portlandite in durability properties is that it could react with the sulfate solution and gypsum. Ettringite is a hexagonal crystal form as classified into hexagonal crystal system, which often exhibits acicular or prismatic shape. The formation of ettringite could develop the strength of cement paste (Neville & Brooks, 2010). Based on this study, C-S-H is observed in two different forms: (i) flocs-like form and (ii) fibrous-like form, as shown in Figure 30A-D. Additionally, the C- S-H is an amorphous or poorly crystalline product, which widely spreads like a network on the matrix of cement paste and enhances durability (Neville & Brooks, 2010) . Evidently, the microstructure of the mix designation of R10 and B5R15 showed the denser matrix occupied by C- S- H, when compared to that of the control mix and mix designation of B10. Moreover, the larger needle- like shape of ettringite was obviously found in mix designation of R10, which provided the greater compressive strength development than the control mix.

The microstructural analysis using SEM images of specimens exposed to sulfuric solution is shown in the Figure 31. The disintegration characteristics and unshaped crystals of the hydration and pozzolanic products were found in all mix, which contributed to the loss in weight and compressive strength. A monoclinic plate of gypsum (G), ettringite (E), and C- S-H was revealed in Figure 31A-D. The loosened matrix by the corrosion of sulfuric acid and formation of gypsum and ettringite could contribute to more pore spaces and some cracks. It can be seen that the microstructure of ternary mix designation of RHA and CB showed the lesser loosed matrix than that of the control mix. This could contributed to the pozzolanic reaction, which produced more secondary C-S-H and lessening the pore spaces.

The SEM images of specimens exposed to sodium sulfate solution are demonstrated in Figure 32. Particularly, the mix designation of R10, B10, and B5R15 revealed the mild deteriorating characteristics of sodium sulfate attack, when compared to the control mix. Accordingly, the dense matrix that was observed in those mix provided the less pore spaces for the formation of gypsum (G), ettringite (E) and microcracks.

As a result, this indicated that the binary mix of RHA up to 30%, CB up to 10%, and the ternary mix of 5%CB and 15% RHA promoted the durability of the POBC mortar due to the densification of microstructure by C- S-H from pozzolanic reaction. This finding is in agreement with Hu et al. (2020), Fernandez et al. (2016), and Mohseni et al. (2017).



Figure 30 SEM images of control mix (A), binary mix designation of R10 (B), B10 (C), and ternary mix designation of B5R15 (D) cured in water for 56 days.



Figure 31 SEM images of control mix (A), binary mix designation of R10 (B), B10 (C), and ternary mix designation of B5R15 (D) after exposure to sulfuric acid solution



Figure 32 SEM images of control mix (A), binary mix designation of R10 (B), B10 (C), and ternary mix designation of B5R15 (D) after exposure to sodium sulfate solution

#### **Properties of POBC concrete**

### **Rapid chloride permeability test (RCPT)**

The RCPT was carried out as per ASTM C1202 for the POBC concrete specimens with different mix designations. The apparatus for RCPT was developed by Suriyachoto & Tonnayopas (2013) in accordance with ASTM C1202. This apparatus consisted of transformer (47 VAC, 13 A), copper circuit board, bridge diode, capacitor, regulator, heat sink and fan, panel voltmeter, and ammeter, as shown in Figure 33A. The alternating current (A.C.) will be input to the apparatus, and it will be transformed to a direct current (D.C.) by full wave bridge rectifier, then the D.C. will be transferred to voltage regulator circuits. The electricity with constant voltage of 60 V was generated on the surface of tested specimen for 6 h. Then, the output of coulombs were measured by ammeter and recorded every 30 mins. The chloride ion permeability of each specimen was classified by the charge passed result. The specimen prepared for the test are shown in Figure 33B. The tested specimens were cured for 28 and 56 days before testing, the total charge passed per coulomb results are presented in Figure 34. Corresponding to ASTM C1202, the criteria for chloride ion penetrability is determined as tabulated in Table 12. The highest 28-day and 56-day chloride ion penetrability were observed in the mix containing 30% CB (7,536 and 6,083 coulombs), while the lowest 28-day and 56-day chloride ion penetrability were found in the binary mix containing 30% RHA (507 and 336 coulombs). Thus, an increase in curing age and RHA decreased the chloride ion penetrability, whereas an increase in CB increased the chloride ion penetrability. The similar trend was observed in the mix designation containing ternary blends of CB and RHA, the very low chloride ion penetrability of 28-day and 56-day specimens was 718 and 598 coulombs, respectively, for the mix designation B5R15 and B10R20, respectively. Hence, the optimum ternary blend ratios of CB and RHA could be considered as 5 to 10% CB with 15 to 20% RHA substitution by weight of OPC in the POBC concrete. Based on the investigated results in this study, a significant decrease in chloride ion penetration was prominently found in the mix supplemented with RHA. The supplementing RHA into CB reduced the chloride ion permeability by 56%, 81%, 76%, 86%, and 76%, respectively, for the ternary mixes of B5R5, B5R15, B15R5, B10R20 and B20R10, compared to the minimum incorporation of 10% CB (mix designation B10) cured for 56 days.
This is due to the greater pozzolanic activity of RHA than CB, which could improve the microstructure by producing more C- S- H and denser the matrix. Correspondingly, previous studies reported that the RHA excellently performed the important role in improving chloride ion penetrability over metakaolin (Gill & Siddique, 2018), sugarcane-bagasse ash and palm oil fuel ash (Joshaghani & Moeini, 2018), and fly ash (Chindaprasirt et al., 2008).

Overall, an increase in curing age up to 56 days with an incorporation of RHA and ternary blends of CB and RHA reduced the chloride ion penetrability of POBC concrete, which were in acceptable range for low to very low chloride ion penetrability, whereas that of the control mix steadily fell in the moderate range base on ASTM C1202.



Figure 33 The apparatus for RCPT (A) and concrete specimen prepared for RCPT (B)



Figure 34 Total charge passed per coulomb from RCPT at different mixes and ages

Total charge passed (coulombs)	Penetrability		
>4.000	High		
2,000 to 4,000	Moderate		
1,000 to 2,000	Low		
1,000	Very low		

Table 12 Criteria for chloride ion penetrability from RCPT according to ASTM C1202

## **Capillary absorption coefficient**

The ability of concrete's surface to absorb water by capillary suction is determined as capillary water absorption. The capillary absorption coefficient is the rate of capillary water absorption, which is a mass gain per unit area of exposed specimen's surface to water as a function of time. The capillary absorption coefficient of POBC concrete with different mix designation at the age of 28 and 56 days was measured and calculated in accordance with ASTM C1585 (2020), as shown in Figure 35. It can be clearly seen that an increase in curing age and percentage replacement of RHA decreased the capillary absorption coefficient. For example, the binary mix containing maximum 30% RHA substitution by weight of an OPC showed the lowest capillary absorption coefficient by 0.06 kg/mm $^2$ /h $^{0.5}$  at the age of 56 days, which was 40% lower than that of the control mix. Conversely, an increase in CB increased the capillary absorption coefficient. The lowest capillary absorption coefficient of the binary mix containing 10% CB was 0.11 kg/mm $^2/\mathsf{h}^{0.5}$  at the age of 56 days, which was 10% higher than that of the control mix. In addition, for the mix designation containing the ternary blends of CB and RHA, the lowest capillary absorption coefficient was found in the ternary mix designation B5R15 and B10R20 cured for 56 days, which were equally 0.07 kg/mm $^2$ /h $^{0.5}$ . Coincidently, the capillary absorption coefficient was in the same trend of the chloride ion permeability. This is due to the capillary pore was filled up by C-S-H, which in turn resulted in the denser matrix. Thus, the chloride ion permeability and capillary absorption coefficient reduced. Moreover, the more  $SiO<sub>2</sub>$  was provided by the more amount of micro RHA particles, which reacted with Ca(OH) $_2$  and generated more C-S-H (Balapour et al., 2017).



Figure 35 Capillary absorption coefficient of different binary and ternary mix designations and curing ages

## **Indirect tensile strength**

The concrete specimens after tested for the RCPT, were then taken to determine the indirect tensile strength in compliance with Brazilian method ( the test method was adapted from ASTM C496/C496M (2017)). The results of indirect tensile strength are in range of 2 to 4 MPa, as shown in Figure 36. It can be seen that the indirect tensile strength increased with prolonged curing age. The development of 56-day indirect tensile strength is clearly found in the binary and ternary mix designation of R10, R20, B10, B5R15, and B10R20, which was 23%, 3%, 3%, 14%, and 7%, respectively, greater than the control mix. On the contrary, the reduction in 56-day indirect tensile strength was observed in the mix designation of R30, B20, B30, B5R5, B15R5, and B20R10, which was 5%, 3%, 4%, 2%, 1%, and 3%, respectively, lower than the control mix. As a result, an increase in CB content decreased the indirect tensile strength as well as the incorporation of RHA content exceeded than 20% by weight of an OPC. The development in indirect tensile strength of the binary mix incorporating the optimum amount of up to 20% RHA and 10% CB could be due to the

pozzolanic reaction of the reactive micro  $SiO<sub>2</sub>$  particles from RHA and CB, which in turn promoted the precipitation of C- S-H and the densification of POBC concrete. The previous work reported that the maximum improvement in tensile strength of 14% was found in the binary mix containing 15% RHA as an OPC replacement, which could ascribed to the improvement of the bond between interfacial transition area of the specimen by C- S- H (Qureshi et al., 2020). This finding also in line with the previous study, that the optimum 10- 15% CB improved the indirect tensile strength of concrete specimen by 5%, compared to the mix without adding CB at the age of 180 days (Masood et al., 2020).



Figure 36 Indirect tensile strength of different binary and ternary mix designations and curing ages

## **Criteria and comprehensive properties of POBC mortars and concretes**

Based on the standard requirement of ASTM C270, the maximum replacement of up to 30% of binary RHA mixture and ternary blends of RHA and CB by weight of OPC can be possibly considered as a practical implication for type S and type M cement mortars and concrete with chloride ion penetrability resistance, as tabulated in Table 13. However, the maximum replacement of 20% of CB for binary mixture is recommended for using as a natural pozzolan without restrain the compressive strength development, as per ASTM C618. Table 13 Criteria and comprehensive properties of POBC mortars and concretes with different binary and ternary mix designations for future practical implication



## **Conclusions and Suggestions**

## **Conclusions**

This study focused on utilization of POBC as fine and coarse aggregate substitution and incorporating binary and ternary mixes of CB and RHA as a partial OPC replacement in green mortar and concrete production. Based on the study, these following conclusions and suggestions can be stated:

1. The physical properties, chemical compositions, and morphology of POBC showed that the POBC can be used to replace natural aggregate for nonstructural mortar and concrete production.

2. The chemical compositions and physical properties of CB and RHA can be classified as type N and type C according to ASTM C618 (2019).

3. The setting times were decreased by an increase in RHA replacement level and a decrease in CB replacement level. Likewise, a decrease in cement contents by an increase in CB and/RHA replacement level reduced the heat of hydration.

4. The flowability of fresh mortars were reduced as an increase in CB and/ RHA replacement level due to the fineness of CB particles and filler effect of RHA. Subsequently, the fresh density of mortars were slightly decreased by an increase in CB and/ RHA replacement level due to the lower true density and specific gravity of CB and RHA compared to that of OPC.

5. The compressive strength increased as a function of curing age. The optimum binary replacement level of CB and RHA were 10% and 20%, respectively. The highest compressive strength was found to be 53 MPa for the mix containing 20% RHA at the age of 56 days. Accordingly, the highest compressive strength of the mix containing 10% CB cured for 56 days was 41 MPa. The optimum ternary replacement level of CB and RHA was the incorporating of 5% CB and 15% RHA, which provided the highest 56-day compressive strength of 48 MPa. However, the compressive strength values of all binary and ternary mixtures were classified into type S and type M mortars, which could be possibly used in both foundation and traditional purpose.

Subsequently, the SAI results were in accordance with the compressive strength developments. Moreover, the 28-day SAI values of binary RHA mixture and ternary blends of CB and RHA were met the standard requirement of ASTM C618 (2019). Thus, the CB and RHA could be utilized as an OPC replacement as up to 20w.% and 30wt.%, respectively, which could provided the SAI not lesser than 75% of the control mix.

6. The water absorption and apparent porosity decreased with (i) prolonged curing ages, (ii) a reduction in CB replacement level, and (iii) an increase in RHA replacement level.

7. The deteriorations due to sulfuric acid and sodium sulfate attack: compressive strength and weight variations, as investigated in this study were mitigated by an increase in RHA replacement level and curing ages.

8. According to the XRD analysis and SEM images, the mix incorporating RHA apparently revealed dense microstructure with C-S-H forming compared to control mix and mix incorporating CB.

9. The rapid chloride ion permeability and capillary absorption coefficient of concretes decreased as an increase in RHA replacement level and curing ages. The very low ranges of total charge passed were found in the binary mix incorporating up to 30% RHA, ternary mix of 5-10% CB and 10-20% RHA.

10. The indirect tensile strength decreased as an increase in CB replacement level, while the replacement level of not exceeded than 20% RHA provided the development in indirect tensile strength.

## **Suggestions**

1. The water reducing agents are recommended to add into the green mortars or concrete containing SCMs or other type of wastes and/ or recycle aggregates in order to develop the high performance in both strength and durability.

2. The CB could be activated by appropriate temperature to improve the pozzolanic activity and induce the amorphousness phases.

3. The investigations of fire resistant should be carried out to consider the suitable usages of these mortars and concretes as well as develop the mix designation and production process.

4. To promote the sustainable construction, reduce the use of natural resources, and degrade the local wastes, the potential of developing a cementless composite containing 100% of palm oil boiler clinker as a natural aggregate substitution is possible, according to the experimental investigation of this study.

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# **APPENDIX A**

## **Proceeding 1**

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## **ENHANCED PROPERTIES OF PALM OIL BOILER CLINKER CONCRETE WITH SANG YOD RICE HUSK ASH**

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Abstract: Laboratory experiments of this investigation are to reclaim palm oil boiler clinker (POBC) as coarse aggregate for concrete and partially replaced Portland cement type 1 with Sang Yod rice husk ash (SYRHA) in proportion of 10%, 20% and 30wt.%. Characterization of POBC aggregate was carried out on grain size distribution and shape analysis, specific gravity, loose and compacted bulk density, water absorption, organic impurity, aggregate impact value and Los Angeles abrasion. Each mixing specimen was kept water to binder ratio at 0.42 throughout this study and varied duration of curing in water for 7, 28 and 56 days. The properties of paste and harden cube concrete in size  $10 \times 10 \times 10$  cm including setting time, bulk density, water absorption and compressive strength and were determined. According to aggregate testing results indicated that POBC was met the specification of normal-weight aggregate. Addition of SYRHA decreased both setting time and hydration temperature of paste. Furthermore, concrete contained 20% SYRHA provided highest 56-day compressive strength of 53.12 MPa and increased water absorption of 5.27%.

Keywords: Palm Oil Boiler Clinker Aggregate, Sang Yod Rice Husk Ash, Pozzolanic, Ecofriendly Concrete

#### **Introduction**

Palm Oil Boiler Clinker (POBC) was a by-product from palm oil mill and it is defined as kind of non-hazardous waste material (Ahmad & Noor, 2007). In the southern part of Thailand, The disposal of POBC has been unidentified and only used for landfill. The origin of POBC was made during the combustion and cooling down process with various condition depended on the mill. For a few decade, POBC were often used as a coarse and fine aggregate replacement in lightweight concrete produced (Mannan & Neglo, 2010; Abutaha, Rasak, & Kanadasan, 2016; Ahmmad et al, 2017; Karim et al, 2017). The mix design of concrete containing POBC was different from others normal aggregate according to their porous and rough surface texture characteristics. Thus, Mannan & Neglo (2010) were investigated the suitable mix design for POBC concrete. They were found that using POBC as coarse aggregate replacement with maximum size at 20 mm including Ordinary Portland cement (OPC), sand, and superplasticizer at w/c ratio 0.48-0.60 and cure in tap water for 28 days. Which can provide the compressive strength in range of 27-35 MPa. Perversely, Abutaha et al. (2016) was produced concrete containing POBC as partial coarse aggregate replacement at 10%, 20%, 40%, 60 and 100%. The compressive strength and hardened density of POBC concrete were obtained in the range of 33–49 MPa and 2074–2358 kg/m<sup>3</sup> at 28 days, respectively. Moreover, it provided the compressive strength to 33 MPa with 100% coarse aggregate replacement by POBC. Thus, POBC has a good potential to replace natural aggregates, making it suitable to



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be used in concrete (Mannan & Neglo, 2010; Abutaha, Rasak, & Kanadasan, 2016; Ahmmad et al, 2017; Karim et al, 2017). According to several researches have been investigated, There are some weak points of using POBC as aggregates that were reported from Abutaha et al. (2017); Ibrahim et al. (2016); Shafigh et al. (2014). The increasing of POBC in concrete as both coarse and fine aggregate reduces the slump and workability in fresh concrete. But only POBC coarse aggregate reduces strength value. Therefore, this research aims to enhance the POBC concrete properties with Song Yod rice husk ash (SYRHA) which was waste of rice husk derived from the rice products from Phattalung, Thailand. It also has a slim grain, dark red pericarp, soft and aromatic of cooked rice as well as nutritional enrich (Banchuen et al, 2009). As wildly usage of rice husk ash (RHA), which is beneficial in many way to concrete i.e. increase the compressive strength and reduce the corrosion deterioration (Gastaldini et al, 2010). In addition, SYRHA was characterized in chemical composition and strength activity index (SAI) of gravel gap-graded aggregate concrete by Tonnayopas & Jitnukul (2013) and met the specification of ASTM C618-12a and D5370-14. Thus, in this experimental investigation to evaluate into suitability of POBC coarse aggregate and SYRHA as a complement for green concrete.

#### Materials and experimental procedures

To carry out this experiment the concrete mix design are following material were used OPC type I according to ASTM C 150, palm oil boiler clinker coarse aggregate (POBCCA) regarded with sieving size between 4.75-12.5 mm, fine aggregate used river sand with fineness modulus of 3.15. The SYRHA powder particles was used as OPC partial replacement at mean particle size 16.7 microns according to Fritsch-Analysette 22 Nanotec laser particle analyser. The particle size distribution of all materials used are illustrated in Figure 1.



Figure 1: Particle size distribution of materials used in this study

### Palm Oil Boiler Clinker Coarse Aggregate

POBC samples in this study were collected from a palm oil factory in Surat Thani, Thailand. It was obtained from the combustion of fibres, seeds and shells of palms after oil extraction process as a fuel for boiler. This combustion process will be taken time for 6-12 h at the range of temperature from 700 to 1200 °C after cooling down. The POBC is 90% greenish grey, 5-10% black and 3-1% pinkish brown color, sintered, vesicular texture with unidirectional and unconnected porous (bubble-shaped localities) because of rapid cooling down, irregular shape,



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brittle edges and exposed in large lumps within 95-70 cm as shown in Figure 2(a). The POBC was washed and air-dried for one week then crushed within size less than 12.5 mm as shown in Figure 2(b).



Figure 2: POBC sample (a) lumps and (b) aggregate used

The chemical composition of POBC was also analysed via X-ray fluorescence (XRF) and tabulated on Table 1. It composed of dominant SiO<sub>2</sub>, and moderate of CaO, K<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub> and MgO contents, respectively. Particularly, loss on ignition (LOI) is very low or negligible. According to microstructure analysis, found that the evidence of POBC occurrence is similarly to "aa" lava or metal slag. The morphology of POBC- under scanning electron microscope (SEM) revealed slightly smooth concave surface with brittle fracture and thermal crack and depicted frame in Figure 3(a). It also observed clearly spherical particles cluster of silica component "Si" (Figure 3(b)).



Figure 3: SEM photomicrograph of POBC (a) slightly smooth concave surface with crack trace and (b) enlargement of silica particles and small void spaces

The aggregate properties of POBC used as coarse aggregate were preliminary tests performed including grain size distribution, specific gravity and water absorption, (ASTM C127-15), loose and compacted bulk density (ASTM C29/C29M-17a), organic impurity (C40/C40M-16), aggregate impact value and Los Angeles abrasion (ASTM C535-16).

### Sang Yod rice husk ash

The Sang Yod rice husk was collected from a local rice mill in Phattalung, Thailand. Then, it was burned into open-air condition by using recycled oil tank for 3-4 days (Figure 4a). Then later it was incinerated in an electrical furnace again at 700 °C for 1 h in order to reduce some



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organic matter (Figure 4b). Then, grinding with jar mill for 12 h and sieved through 325 sieve mesh to obtain the final binder sample and kept in a container to prevent it from moisture. Its chemical composition result was analysed by XRF technique shown in Table 1.

Table 1: Chemical composition of POBC and SYRHA used for this study

Oxides (wt.96)	SiO <sub>2</sub>						AhO3 Fe2O3 MgO P2O5 SO3 K2O CaO MnO Others LOI'	
<b>POBC</b>			65.23 1.66 1.78 5.37 5.50 0.04 9.56 10.00 0.17 0.59					0.04
SYRHA 93.83		0.66				0.36 0.36 1.04 0.21 1.77 0.76 0.11	0.23	0.67



Figure 4: SYRHA after treatment processing of (a) open-air burned and (b) incinerated at 700 °C for 1 h

According to XRF analysis, the amount of  $SiO<sub>2</sub>+Al<sub>2</sub>O<sub>3</sub>+Fe<sub>2</sub>O<sub>3</sub>$  is greater beyond 70% and LOI is also less than 6%. In consequence, the SiO<sub>2</sub> contents of SYRHA is higher than Tonnayopas & Jitmukul (2013) due to the incinerated treatment process reduced unburned carbon. The XRF analysis also confirmed that SYRHA was met the threshold of artificial pozzolans in burnt materials type but cannot identify in any classes although its chemical composition was same as pozzolan definition but its evidence of occurrence was not according to ASTM C 618 and ASTM D 5370. Therefore, SYRHA was conformed to specified requirements of blended poznolan for use in concrete and should be added to brand new type "RHA" of organic pozzolan in opportunity. The microstructure of SYRHA powder was analysed via SEM and can be noticed that the physical characteristic of SYRHA was a spherical shape and almost round (Figure 5(a)). Most particles size were in the range of 2.3-38 microns as shown in Figure 5(b), it effected on more surface area as observed on SEM image.



Figure 5: SEM photomicrograph of SYRHA (a) clusters and (b) spherical and rounded particle of silica



#### Specimen preparation and mix proportions

Whole investigated specimens were cast in cube with dimension of 100×100×100 mm. Then, the concrete mix designs were inspection on ACI 211.1-91 method to approach slump value between 35-50 mm and compressive strength at 35 MPa in 28 days as tabulated in Table 2. Mix proportions of specimens were designed by replacing OPC with SYRHA at 10, 20, 30wt.% and kept water to binder ratio at 0.42 throughout this study. The test specimens were stored in moist air for 24 h and after this period each specimens are marked and removed from the moulds then kept submerged in tap water until  $7, 28$ , and 56 days and taken out to prior test, The various tests were carried out on paste and harden concrete specimens with standard procedures in ASTM specifications containing setting time ASTM C403/C403M-16), heat of hydration (ASTM C186-17), bulk density and water absorption according to ASTM C642-13, and compressive strength modified on ASTM C39/C39M-18.

Table 2: Mix batches of specimens

No.	<b>Mix designation</b>	w/b ratio	Mix containing (kg/m <sup>3</sup> )				
			OPC	Sand	<b>POBCA</b>	<b>SYRHA</b>	
	Control	0.42	500	1315	700	0	
2	<b>SYRHA10</b>	0.42	450	1315	700	50	
3	<b>SYRHA20</b>	0.42	400	1315	700	100	
	<b>SYRHA30</b>	0.42	350	1315	700	150	

#### **Results and discussions**

The result of POBC coarse aggregate testing were tabulated in Table 3. It has coincided with several previous researches results even its physical appearances was not similarly i.e. color, texture but it has the highest specific gravity and lowest 24 h water absorption when compared to previous researches which are in the range of 1.67-1.8 and 2.7-5.4% respectively. (Ahmad & Noor, 2007; Abutaha, Rasak, & Kanadasan, 2016; Abutaha et al, 2017; Ahmmad et al, 2017).





#### Setting time and hydration temperature of paste

The initial and final setting times of OPC partial replacement by SYRHA at 10, 20 and 30% were tested gather with heat monitoring. The increasing SYRHA replacement effected directly on water demand due to ultrafine particles. The paste which contains high content of SYRHA signify less consistent than without SYRHA or control mix since after mixing with the same amount of water as shown in Figure 6(a). The Vicat penetration depth indicated that the increasing replacement level of SYRHA played the key role to decrease both initial and final setting time of paste due to pozzolanic reaction, high water demand and specific surface area. At 30% SYRHA replacement reduced initial and final setting time of paste down to 32% and 16% respectively when compared the result of control mix as depicted in Figure 6(b).



Figure 6: Effects of SYRHA partial replacement on (a) development of setting time behaviour and (b) interval duration of setting time

The hydration temperature of paste also decreased when increased replacement level of  $SYRHA$  due to reduce cement contents or tricalcium silicate  $(C<sub>3</sub>S)$  and aluminate hydrated (C<sub>3</sub>A) by SYRHA as shown in Figure 8. Concordantly, there are 3 factors controlled the heat of hydration including pozzolanic reaction, tricalcium silicate hydrated (C<sub>3</sub>S) content and tricalcium aluminate hydrated content (Hewlet, 2003).



Figure 8: Effects of SYRHA on hydration temperature of paste

### **Bulk density**

The POBC concrete at the ages of 56-day without SYRHA as a cement replacement can be achieved bulk density at 2205.81 kg/m<sup>3</sup> (Figure 9) which is not significantly different from normal weight concrete densities owing to higher specific gravity of POBC coarse aggregate



than previous researches (Mannan & Neglo, 2010; Abutaha, Rasak, & Kanadasan, 2016; Ahmmad et al, 2017). The entire results have shown that increasing replacement level of SYRHA decreased the bulk density at all curing periods. As the result of OPC was replaced by SYRHA, bulk density of concrete also reduced. Conversely, the long term curing ages effected on increasing POBC concrete density by given more hydration process and condensation of concrete structure (Locher & Richartz, 1974).



Figure 9: Effects of SYRHA on bulk density of POBC concrete at different ages

#### **Water** absorption

According to Figure 10, it can be represented that at 10% SYRHA replacement of OPC decreased water absorption of POBC concrete compared to control mix. It is represented the optimum usage for reduce porosity of POBC concrete as water absorption value calculated from the saturated water was taken place on void in concrete. Thence, further water absorption was gone beyond 10% SYRHA replacement of cement and depended on curing time in this study. In general, adding some pozzolans i.e. rice husk ask, fly ash and silica fume can be decreased pore size of concrete. In conversely on this study, due to SYRHA is particle usually absorption water, thus it may not be optimum for pozzolanic reaction and should not over grinding if using SYRHA to produce concrete for durability usage.



## **Compressive strength**

Effect of OPC replacement by SYRHA on compressive strength was depicted Figure 11. All level of replacement performed higher strength at prolonger curing ages. At 20% SYRHA, the compressive strength of 56-day concrete was the highest of 53.12 MPa. The maximum strength



activity index (SAI) of 7, 28 and 56-day concrete were 116%, 113%, 109% at 10% and 20% SYRHA replacement, respectively. It can be implied that at 10% SYRHA was suitable for early strength development and at 20%SYRHA for late strength development since 28 days of curing ages. It can be noticed physical and mechanical properties of POBC concrete blended SYRHA had closely relationships.



#### **Conclusions**

According to experimental results on this study can be delineated the POBC is classified into normal-weight aggregate and was available as coarse aggregate on concrete produced for other construction applications except heavy duty and pavement wearing surfaces. Chemical composition of SYRHA was met the specification of artificial pozzolans in burnt materials type. It is also reduced setting time, heat of hydration of paste and bulk density of POBC concrete. Considering as enhanced POBC concrete, 10% SYRHA replacement of OPC was significant developed the early strength and reduced water absorption of POBC concrete, benefit of 20% SYRHA replacement of OPC was provided satisfactory strength for long term of the curing ages of 28-day concrete.

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## **APPENDIX B**

## **Proceeding 2**

The 13th International Conference on Mining, Materials, and Petroleum Engineering (CMMP 2019), June 13-14, 2019, Krabi, Thailand,

Kueaket, K., & Tonnayopas, D. (2019). Palm oil boiler clinker properties used as a green aggregate for construction. *Proceeding of the 13th International Conference on Mining, Materials, and Petroleum Engineering (CMMP 2019)*, Krabi, Thailand, 86-91.

The 13<sup>th</sup> International Conference on Mining, Materials and Petroleum Engineering (CMMP2019) June 13-14, 2019, Krabi, Thailand

## PALM OIL BOILER CLINKER PROPERTIES USED **AS A GREEN AGGREGATE FOR CONSTRUCTION**

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#### **Abstract**

This research are aimed to characterize the chemical and physical properties of POBC for construction material usage. The physical properties of POBC were tested including grading, fineness modulus, flakiness and elongate index, loosed and compacted bulk density, porosity, moisture content, water absorption, organic impurity, aggregate impact value, sodium sulfate soundness and Los Angeles abrasion. The chemical composition of POBC was analyzed by X-ray florescence technique. Moreover, microstructure of POBC was observed via scanning electron microscope. As the results found that the POBC was meant to be normal weight aggregate. The physical properties of POBC were also indicated that it can be used for concrete production. In addition, compressive strength of POBC concrete and mortar with constant 0.42 water to cement ratio were also found the 56-day POBC concrete and mortar can be provided the compressive strength up to 48.86 and 47.13 MPa.

Keywords: Palm Oil Boiler Clinker, Green Aggregate, Physical Properties

#### **Introduction**

The south part of Thailand are well-known for oil palm cultivating. The products of oil palm also have taken to process in the local mill to get palm oil. The palm oil extraction process has left many waste such as seed, shell, fiber etc. There are one part of the oil extraction process can reduce the fresh oil palm waste by turning the waste to fuel for boiler. The wastes were burned with high temperature and under pressure condition. The extensive burning and imminent cooling process of the stale shell and fibrous of palm oil inside boiler will originate the hardened and compacted materials call "Palm Oil Boiler Clinker" or POBC. Their sinter produces are greenish gray with some dark pink spot in color, unconnected vesicular texture and lightweight. The POBC was widely studied in Malaysia as a lightweight aggregate for concrete production. According to several previous studies, the POBC are often mentioned as a lightweight aggregate and only considering in term of lightweight aggregate replacement at different ratios [1]. Furthermore, the POBC properties were revealed by many Malaysian researches. Their chemical and physical properties can be varied according to the heterogeneous in compacted texture or others burning and cooling environmental conditions. The POBC samples used in this study are also different from others literature reviews in details of color, texture and their appearance. Particularly, Ahmmad et al., Ahmad et al., and Mannan and Neglo [2-4] gained the physical properties of POBC were carried out that their specific gravity was in the range of 1.7-2.2, density was in the range of 683-1,813.23 kg/cm<sup>3</sup> and water absorption was in the range of 1.79-2.67% Investigation using POBC as a coarse aggregate [3] were found the effective mix design for POBC concretes which were used POBC as coarse aggregate and mixed with OPC, river sand and super plasticizer at w/c ratio 0.48-0.60. Their mix design can be provided the 28-day compressive strength of POBC concrete in the range of 27-35 MPa. In addition, Abutaha et al. [1] used POBC as coarse aggregate for concrete also but their mix proportion were design by (DOE) method [1] and without adding water reducing agent. The compressive strength of 28-day POBC concrete were up to 33 MPa. Likewise, considered using POBC as fine aggregate, [6] replaced river sand with POBC in proportion of 5%, 10%, 15%, 20% and 25wt.% kept water to cement ratio at 0.45. It indicated that at beyond 20% replacement can cause the reduction in workability due to the less water absorption of POBC than normal fine aggregate. It may reduce the quality of mortar by formation of void and capillary cavity in mortar matrix due to low workability. Therefore, in Thailand, there were found POBC identically but still were not delineated for waste utilization. Meanwhile, this

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research is focused on physical and chemical properties of POBC obtained from Thailand and the investigation on compressive strength of POBC concrete and mortar were contained a plain POBC coarse or fine aggregates.

## **Materials and methods**

## **Palm Oil Boiler Clinker**

POBC samples were collected from palm oil mill at Suratthani province, Thailand. As observation of morphological features. It is looked likely between crushed POBC and vesicular basalt collected from Buriram province (Figure 1). If considering only texture that were shown in Figure 1. In addition, the chemical composition of POBC [16] (Table 1) were mostly consist of SiO<sub>2</sub> 65.23%, CaO 10.00% and K<sub>2</sub>O 9.56%. Which were identically after Ahmmad et al., Ibrahim and Razak., and Karim et al. [1, 6, 7]





Figure 1 Comparison between (a) Large Lump of POBC and (b) Vesicular Basalt

#### **Experimental testings**

The POBC process generating might be as same as the origin of extrusive igneous rock. Aftermath palm oil extraction process was mill. The fibers and shells were left over. Then, it were taken to use as a fuel to generate the power for boiler. The combustion of remnant palm oil inside boiler will be operated under high pressure and temperature conditions for 16-18 h consecutively. After that it will be suddenly cooled down and became compacted and hardened. Before testing, the POBC were crushed into coarse aggregate (POBCCA) (> 4.75 mm) and fine aggregate (POBCFA) (< 4.75 mm) as shown in Figure 2. The aggregate properties of POBC were carried out in



Figure 2 Crushed POBC Used as (a) Coarse Aggregate (POBCCA) and (b) Fine Aggregate (POBCFA)

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grading, fineness modulus, flakiness and elongate index, loosed and compacted bulk density, moisture content, water absorption, organic impurity, aggregate impact value, sodium sulfate soundness and Los Angeles abrasion were carried out according to ASTM test designation as tabulate in Table 2.

#### Table 2 Physical Properties Testing Designation



#### Preparation of concrete and mortar

Ordinary Portland cement, type 1 were used to mix with coarse and fine POBC. The POBC coarse aggregate concrete (POBCCA-Concrete) were investigated in the size of cube with dimension 100×100×100 cm including with river sand, fineness modulus 3.15. The POBC fine aggregate mortar (POBCFA-Mortar) were investigated in the size of cube with dimension  $50 \times 50 \times 50$  mm. The POBC concrete and mortar mix proportion were calculated and designed according to ACI method [15] to achieve 28-day compressive strength of 35 MPa by using constant 0.42 water to cement ratio as controlled factor, due to the compressive strength of concrete or mortar was inversely correlated with water content of mix proportion [17]. Furthermore, the water to cement ratio must be appropriate with the maximum size of POBCCA as 10 mm and fineness modulus of POBCFA as 3.74 that were conformed to ACI mix design method [15, 17]. Both POBC concrete and mortar were cured in tap water at ambient temperature (25 $\pm$ 5 °C and relative humidity 70-85%). The mix proportion of POBC concrete and mortar were tabulated in Table 3.

Table 3 POBC Concrete and Mortar Mix Design in This Study



#### **Results and discussions Microstructure of POBC**

The microstructure of crushed POBC were found that it is large irregular shape Figure 3 a) and slightly smooth surface with some concave solidify fracture at the edge as was shown in Figure 3 b). Although many pores were observed when the POBC was not crushed as a bumpy surface. But literally, the photomicrograph images reveals that the POBC surface texture was smooth and rarely micro-pore with high surface area caused it contains lots of  $SiO<sub>2</sub>$  sintered amorphous particles. At figure 3 b) on the top right corner, there were largely conchoidal fractures

similarly to quartz assumed that caused by rapid solidification process along shortly cooling down.

#### **Gradation**

Crushed POBC as a coarse aggregate or POBCCA was partially in the range between upper limit and lower limit. The fineness modulus of POBCCA is 5.58 which mean the maximum size of POBCCA is 12 mm and the

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average particle size is 10 mm. Illustration shown in the red rectangle in Figure 4, The POBCCA was not well-graded and each of particle size was largely uniform grading curve. Nevertheless, using POBCCA for concrete must add sand to fill the void between poor-graded POBCCA particles and require more cement content due to poor-graded aggregate need more water to cement ratio. On the other hand, crushed POBC used as a fine aggregate or POBCFA was also fell partially into the range of upper limit and lower limit. The fineness modulus of POBCFA is 3.74 which is meant as a coarse sand. As shown in the blue line in Figure 4, the POBCCA was not well-graded and contained more coarse sand size. Which require more cement and water contents also. Additionally, both POBCCA and POBCFA are quite angular and irregular in shape.



Figure 3 SEM Photograph of Crushed POBC (a) Irregular Shape [16] and (b) Magnification of Surface



Figure 4 Grading Curve of POBCCA and POBCFA

#### **Physical properties**

Based on the testing result as tabulated in Table 4, the crushed POBC was not met in lightweight aggregate requirements specification on their specific gravity, density and weight. Moreover, porosity of crushed POBC was also low as approximately 5.59%. Percentage of voids in both loose and compacted bulk density were 39.66% and 35.68%, respectively which were represented of angular particle characteristics. The crushed POBC was rather flat after crushing process. The flakiness index of crushed POBC was in the high value as 66.62%. Which was gone beyond the requirement for 2 times. The flat or flaky particle would need more water and cement contents and caused bleeding as well. Although, the crushed POBC was satisfy in elongate index as 8.91%. The 1-and 7-day water absorption of crushed POBC were also low as 1.42% and 3.00%. Moreover, these kind of POBC was not contained harmful organic impurity. The sodium sulphate soundness test was also carried out for 5 cycles and found that it was 2.05% weight loss indicated high soundness. The aggregate impact value and loss angles abrasion were defined as the crushed POBC can be used for concrete.

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### **Compressive strength**

Compressive strength of both POBCCA concrete and POBCFA mortar were performed beyond the basic requirement designed by ACI method [15] at the age of 28-day which was 35 MPa. The compressive strength of 7day POBCCA concrete and POBCFA mortar was 32.53 and 21.36 MPa, respectively. Both was increased approximately 20% and 33% after the age of 28-day and 56-day, respectively. Hence, the compressive strength of 28-and 56-day POBCCA concrete and POBCFA mortar were provided 39.50 MPa, 39.82 MPa, 48.86 MPa and 47.13 MPa, respectively. It shown that curing aging was effected to develop compressive strength of POBC specimens.



Figure 5 Compressive Strength of (a) POBCCA Concrete and (b) POBCFA Mortar at Different Curing Ages

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### **Conclusions**

According to the physical properties and morphology analysis under scanning electron microscope, it can be identified that POBC was a normal weight aggregate. The characteristics of shape and texture of POBC were angular and flaky after crushing. Thus, utilization of POBC as an aggregate must be given precedence to the suitable water and cement contents in order to get the best POBC concrete or mortar quality. Moreover, based on the tested results, the POBC aggregate is recommend for eco- or green- concrete works which is not satisfied into heavy duty, structural or surface pavement. The compressive strength of 56-day POBC concrete and mortar, obtained from this investigation, were 48.86 MPa and 47.13 MPa respectively. Hence, POBC aggregate can be the alternative green aggregate source using for eco-concrete application as well.

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### **APPENDIX C**

## **Research article 1 (Indexed by Scopus)**

Kueaket, K., & Tonnayopas, D. (2021). Compressive strength and durability performance of mortar containing palm oil boiler clinker aggregate, rice husk ash, and calcium bentonite. *Journal of Applied Engineering Science*, 19(1), 193-203. doi: 10.5937/jaes0-27580



**Original Scientific Paper** 

Istraživanja i projektovanja za privredu Journal of Applied Engineering Science

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Paper number: 19(2021)1, 781, 193-203

### **COMPRESSIVE STRENGTH AND DURABILITY** PERFORMANCE OF MORTAR CONTAINING PALM OIL BOILER **CLINKER AGGREGATE, RICE HUSK ASH, AND CALCIUM BENTONITE**

### Kamolchanok Kueaket, Danupon Tonnayopas\*

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The utilization of local waste by-products as a building material has attracted great attention for an environmental sustainability and become a fundamental part of sustainable construction. In this experimental research, the local palm oil industrial waste and agricultural waste are utilized for the green mortar production. To examine the compressive strength and the durability performance of the green mortar mixtures, Palm oil boiler clinker (POBC) was used as a substitution material for natural fine aggregate. An ordinary Portland cement was partially replaced by rice husk ash (RHA) and calcium bentonite (CB) in the proportion of 10%, 20%, and 30% by weight of cement. The compressive strength, water absorption, porosity, durability against sulphuric acid and sodium sulphate attacks, and microstructures of the POBC mortar mixtures were evaluated at the curing age of 7, 28, and 56 days. The experimental results revealed that the compressive strength, water absorption, porosity, and durability characteristics of POBC mortar incorporating RHA and CB were improved by long-term curing. Particularly, the 56-day POBC mortar incorporating up to 30% of RHA and 10% of CB yielded the superior durability against sulphuric acid and sodium sulphate attacks.

Key words: palm oil boiler clinker, rice husk ash, calcium bentonite, compressive strength, durability against sulphuric acid and sodium sulphate attacks

### **INTRODUCTION**

The exploitation of waste materials in mortar and concrete is studied as a means toward a sustainable, environmental-friendly construction [1]. Currently, the practice of using the industrial by-products as supplementary cementitious materials (SCMs) and as an aggregate substitution is spreading constantly. Apart from strength properties, durability is an important issue for local mortar production. Several studies reported that the introduction of the SCMs from waste by-products such as fly ash, silica fume, ceramic waste, ground granulated blast-furnace slag, and rice husk ash can improve the long-term durability of the cementitious composite. This is due to the formation of calcium silicate hydrate by the pozzolanic reaction which improves the pore structure and clogs up the cement matrix [2-6].

The palm oil boiler clinker (POBC) is a waste-by product of palm oil mill [7]. In 2019, the agricultural productivity of palm oil in Thailand was 16.8 million tons [8]. Particularly, the vast cultivation area of palm trees covers 6,765 km<sup>2</sup>, with an approximate annual crude palm oil production of 2.4 million tons [9]. A solid wastes of crude palm oil production (such as palm fibers, empty fruit bunch, palm shells) are used as biomass fuels in boiler for sterilization process [10]. The POBC and palm oil fuel ash (POFA) are derived after the end of combustion in boiler. The POBC, POFA, and several industrial wastes derived from the palm oil industry have resulted in a significant environmental impact and landfill usage [11-12]. In an effort to mitigate this, the POBC properties were tackled by various researchers. The study on the feasibility of using the waste of palm oil mill for construction applications confirmed that the utilization of palm oil clinker as a sand replacement can reduce the cost of construction and carbon emission by 17% and 9.6% compared to that of using natural river sand [13]. In addition, the use of high replacement ratio of palm oil clinker as a sand substitution caused a 56-day mortar's strength reduction of only 22%, as compared to conventional sand mortar [14].

Rice husk ash (RHA); an agricultural by-product, is obtained after using the rice husk as a local biomass fuel. The main constituent of RHA is the reactive amorphous SiO<sub>2</sub> [15], therefore, several studies investigated its contribution to the properties of cementitious materials when used as an additive. It was confirmed through an XRD analysis that the addition of reactive SiO<sub>2</sub> derived from RHA reacted with the Ca(OH)<sub>2</sub> contents of the hydration products [16]. Thus, consuming a portion of the free Ca(OH), and forming the additional calcium silicate hydrate (C-S-H) gel. Moreover, it was found that the pozzolanic reaction was promoted by the addition of RHA [17-181. The promoted pozzolanic reaction contributed to the strength development and increased C-S-H content. As a reactive filler, C-S-H plays an important role in reducing the capillary pore size in the cement paste. Moreover, utilizing the RHA as supplementary cementitious material can enhance the acid resistance of recycle-aggregate concrete by decreasing calcium hydroxide (Ca(OH)<sub>2</sub>) content, while strengthening the bond between the dif-

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ferent phases within microstructure [19]. This explains the mortar's enhanced durability by the additional C-S-H, since the C-S-H forms by cement hydration and pozzolanic reaction. The deterioration reaction between the hydration product of the cement paste and sulphuric acid is the depletion of Ca(OH), and the forming of gypsum (CaSO ..2H<sub>2</sub>O). Then, the degradation mechanisms by sulphuric acid attack occur. Subsequently, the gypsum gel can fill up the pore, intensive gypsum deposit, and the pore structure damaged [20-21].

Furthermore, calcium bentonite (CB) is a natural pozzolanic material and contains more than 60% of SiO<sub>2</sub> [22]. According to the assessment of the strength activity index (SAI) of concrete containing CB, the addition of up to 25% CB by weight of cement can provide a satisfactory SAI (more than 75%). Likewise, the compressive strength of concrete containing 15% CB by weight of cement is close to that of control concrete at the age of 28 days (the strength reduction was only 5% in comparison with the compressive strength of the control concrete specimen) [23]. In addition, the longer curing periods have a positive impact on improving the strength and durability characteristic of the mortar containing CB [24]. Besides, the addition of 8% CB decreased the porosity of 28-day mortar by 16.9% [25-26].

In the pursuit of promoting sustainable construction, this research focuses on the utilization of local by-products as both a fine aggregate substitution and a cement replacement. Therefore, in this study, the RHA and CB were introduced as a partial cement replacement, along with a 100% POBC fine aggregate replacement to determine the strength and durability characteristics of the mortar mixtures at different curing ages. The strength and durability characteristics; namely, the compressive strength, porosity, water absorption, and resistance to mild concentration of sulphuric acid and sodium sulphate attack were investigated together with the microstructural characterization via scanning electron microscope (SEM).

### **MATERIALS AND METHODS**

### **Materials**

An ordinary Portland cement (OPC) complying with ASTM C150 [27], was used in this study, while the RHA derived after raw rice husk (a local by-product from Phattalung province, Thailand) was conventionally burned in open air as a local biomass fuel. Moreover, the calcium bentonite (commercial grade) was purchased from a manufacturer in Saraburi province, Thailand. Meanwhile, the POBC was collected from the waste disposal area near the palm oil plant at Surat Thani province, Thailand.

Firstly, the obtained RHA was then exposed to a temperature of 700°C for 1 hour in a muffle furnace in order to eliminate some presence of unburned carbon and/or some organic matter contaminants. Then, it was ground with a jar mill at 70 rpm for 12 hours. The particle size distributions of the RHA and CB were analyzed by a laser particle analyzer (LPSA); model: Analysette 22 Nano tec. The mean particle size  $(d_{so})$  of RHA and CB was 12.4 and 2.5 µm, respectively. Regarding the POBC, it was washed to remove any clay particles and organic matter. Then, it was oven-dried at 105±5°C for 24 hours, to be crushed with jaw crusher and sieved through No.4 sieve mesh (or aperture size of 4.75mm). The fresh POBC samples obtained from palm oil mill disposal area, crushed POBC, RHA, and CB powders are depicted in Figure 1. The chemical composition of the POBC, RHA, and CB were analyzed via a wavelength dispersive X-ray fluorescence spectrophotometer (WDXRF); model: Zetium. and it is expressed in Table 1. The total amount of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>2</sub>, and Fe<sub>2</sub>O<sub>2</sub> contents of RHA and CB were higher than 70%, which met the chemical requirements of class N (raw or calcined natural pozzolans) and class F (fly ash) as per ASTM C618-19 [28]. The percentage passing results of the sieve analysis for crushed POBC, RHA, and CB were plotted against particle diameters and presented in Figure 2. Finally, the physical proper-



Table 1: Chemical composition of POBC, RHA, and CB analyzed by WDXRF



Figure 1: Lumps of POBC collected from palm oil mill disposal area (a), Crushed POBC (b), RHA powder (c), and CB powder (d)

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ties of POBC fine aggregate were tested and tabulated in Table 2 for convenience.

### **Mix proportions**

Seven mortar mixtures were prepared with POBC fine aggregate and incorporating RHA and CB as an ordinary Portland cement (OPC) replacement. The OPC was partially replaced by RHA and CB at the replacement ratios of 10%, 20%, and 30% (by percent weight of OPC), respectively. The mixture proportions are 1:2.75 (Binder: POBC aggregate). Water to binder ratio of the mixture was kept constant at 0.48 in accordance with ASTM C109 [29]. The percentage of flow was carried out as following ASTM C 1437-15 [30]. The mixture series are tabulated in Table 3.

### **Testing methods**

To determine the compressive strength, water absorption, porosity, and durability against acid and sulphate attacks, three cube specimens of 50-mm of each mixture were cast and then cured in saturated lime water at the temperature of 27±5°C for a period of 7, 28, and 56 days before testing. The compressive strength test was performed according to ASTM C109 [29] by using digital concrete compression machine model KC-150g with capacity 1,500kN. The strength activity index (SAI) was calculated as per ASTM C618-19 [28]. The water absorption and porosity measurements were conducted in accordance with the Archimedes method based on the ASTM standard C373-14a [31].

The sulphuric acid (H<sub>2</sub>SO<sub>2</sub>) and sodium sulphate (Na<sub>2</sub>SO<sub>2</sub>) resistance tests were carried out after the specimens were cured in saturated lime water for periods of 7, 28, and 56 days. During testing, the 7-day, 28-day, and 56-day specimens of each mixture were weighted by digital precision balance (0.001g), and then exposed to 0.005M ( $pH \approx 2.5$ ) sulphuric acid solution (H<sub>2</sub>SO<sub>4</sub>) and 0.5M (pH≈8) sodium sulphate solution (Na<sub>2</sub>SO<sub>4</sub>) for a period of 42 days. The





### Table 2: Physical properties of crushed POBC (fine aggregate)





Figure 2: Grading analysis of raw materials

acid and sulphate solutions were refreshed every week, in order to maintain the pH value and reduce the effect of pH gaining by excessive lime due to the cement hydration. After 42 days of exposure to each acid and sulphate solutions, the weights and compressive strengths of each specimen were examined. Subsequently, through comparing the weight before and after exposure to the acid and sulphate solutions, the weight loss was estimated. Moreover, the change in compressive strengths after exposure to acid and sulphate solutions was investigated in comparison to the compressive strength of the specimen before exposure, as to assess the compressive strength loss.

For the microstructural analysis of the POBC mortar's cement paste, the scanning electron micrographs were carried out by using Thermo Fisher Scientific scanning electron microscope, model Quanta 400 (SEM-Quanta) at the accelerating voltage of 20 kV.

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### **RESULTS AND DISCUSSIONS**

### **Compressive strength**

The compressive strength results of the POBC mortars incorporating the RHA and CB at different ages are presented in Figure 3. Generally, it is evident that the 7-day compressive strengths of the RHA and CB specimens were lower than the control specimen. However, it can be noticed that the compressive strength of all mix types increased through a prolonged period of curing.

Particularly, it was observed that the mortar incorporating the RHA developed strength gradually in the long term. At the curing age of 56 days, an increase in the compressive strength and strength activity index (SAI) was 16%. 18%, and 15% for the mortar specimens incorporating 10%, 20%, and 30% RHA, respectively, as compared to that of the control mortar; This is shown in Figure 3(a) and 4. This increase in the compressive strength of RHA mixtures with age could be due to the high pozzolanic reaction that is gradually promoted by the reactive amorphous SiO2 contents of the RHA [32-33].

On the other hand, the compressive strength decreased with the increase in the CB replacement ratio. At the age of 56 days, a decrease in the compressive strength of the CB mixtures was observed to be 9% 15% and 20% for the mortars incorporating CB content of 10%, 20%, and 30%, respectively; as shown in Figure 3(b). However, the





Figure 3: Compressive strength of POBC mortar containing RHA (a) and CB (b) at different ages

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Figure 4: Strength activity index of POBC mortar containing RHA and CB at different ages

percentage of the strength development from the age of 28 days to 56 days for the specimens incorporating 0% and 10% CB was 26% and 39%, respectively. Besides, the strength activity indexes (SAI) of the mixture containing 10% CB content were 81%, 83%, and 91% at the ages of 7, 28, and 56 days, respectively; as shown in Figure 4

Thus, it is observed that the replacement by weight of OPC by 10% CB provided the lowest strength reduction and the most desirable SAI, as per the standard requirement of ASTM C618-19 [28]. This finding is in good agreement with the previous studies [24, 34].

### Water absorption and porosity

Figure 5 shows the water absorption and porosity of the POBC mortars incorporating RHA and CB at the age of 7, 28, and 56 days. In general, the water absorption and porosity of all mixtures decrease as the curing age increases

An increase in the replacement ratio of the RHA significantly reduced the water absorption and porosity of the RHA specimens, compared to that of the control specimen; as shown in Figure 5(a). At the age of 56 days, both the water absorption and porosity of the specimen incorporating 30% RHA were lower than the control specimen by 36% and 33%, respectively. The decrease in porosity could be due to the high pozzolanic reaction and the filler effect of RHA [35].

On the other hand, it was observed that incorporating 10% CB content reduced the water absorption and porosity by 9% and 14%, respectively, compared to that of the control specimen at the curing age of 56 days; as shown in Figure 5(b). However, an increase in CB replacement of OPC above 10% (by weight) showed a negative impact on the water absorption and porosity characteristics of the mortar. This finding is consistent with previous researches [25-26], where it was shown that incorporating 8-15% CB content can decrease the porosity of mortar. Conversely, they pointed out that the water absorption and porosity of the mortar incorporating

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high replacement ratio of CB (more than 15%) increased owing to the high CB content, which could hinder the hydration reaction. Thus, it could lead to aggravating the more heterogeneous matrix structure of mortar. In addition, an excess CB replacement absorbs additional water content. Consequently, the water absorption of the mortar incorporating a high amount of CB increases [23].

### **Effects of sulphuric acid attack**

### **Weight loss**

The weight loss of the specimens containing RHA and CB at different ages was assessed after 42 days of exposure to 0.005M (pH=2.5) sulphuric acid solution, as shown in Figure 6. The overall trend shows a decrease in weight loss with prolonged curing before exposure to sulphuric acid.

The examination of the RHA specimens, clearly demonstrates that an increase in the RHA replacement ratio significantly reduced the weight loss of the RHA specimens compared to that of the control specimen. For instance, the weight loss was 1.53%, 0.81%, 0.68% and 0.47% for the 56-day specimens containing 0%, 10%, 20% and 30% RHA content; as shown in Figure 6(a).



Figure 5: Water absorption and porosity of POBC mortar containing RHA (a) and CB (b) at different ages

 $(b)$ 



Figure 6: Weight loss of POBC mortar containing RHA (a) and CB (b) after exposure to sulphuric acid solution

Likewise, it was found that the 56-day specimen containing 10% CB content, provided 0.97% of weight loss, which was lower than that of the control specimen and the specimens containing 20% and 30% CB content; as per Figure 6(b). On the contrary, an increase in the CB replacement ratio of more than 10% performed greater weight loss and has a negative effect on the resistance to sulphuric acid exposure.

### **Strength loss**

The compressive strength loss values of different mixtures at the curing ages of 7, 28, and 56 days are revealed in Figure 7. Generally, a decrease in the strength loss is noted with an increase in the curing age before the exposure to sulphuric acid. For instance, the strength loss was recorded to be 4.93%, 3.84%, 2.90% and 1.59% for the 56-day specimens incorporating 0%, 10%, 20% and 30% RHA, respectively, as depicted in Figure 7(a). In addition, the 56-day specimen containing CB content of 10% provided the lower strength loss (4.43%), in comparison with the specimen containing 20% and 30% CB content; with a strength loss of 5.54% and 6.08% (Figure  $7(b)$ ).

Consequently, an increase in RHA replacement ratio of RHA showed a decrease in the strength loss and has a positive effect on sulphuric acid resistance, while the specimen incorporating more than 10% CB content provided an increase in strength loss and has an adverse

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effect on the resistance of POBC mortar to sulphuric acid attack.

### Effects of sodium sulphate attack

### Weight gain

An increase in weight was observed in all specimens after 42 days of exposure to 0.5 M (pH=8) sodium sulphate solution, as demonstrated in Figure 8. Generally, there is a mild decrease in the weight gain with exposure to sodium sulphate solution after prolonged curing. For example, the weight gain was 0.13%, 0.09%, 0.07% and 0.03% for the 56-day specimens incorporating 0%, 10%. 20% and 30% of RHA, respectively, as presented in Figure 8(a). Additionally, it is found that the 56-day specimen incorporating 10% CB provided the lowest weight gain by 0.12%, whereas the weight gains of the control specimen and the specimens with 20%, and 30% CB content were 0.13%, 0.15%, and 0.16%, respectively, as illustrated in Figure 8(b). Hence, the increase in proportions of RHA replacement of OPC and the specimen incorporating 10% CB yielded a better performance to sulphate resistance by demonstrating the lower weight gain when compared to that of the control specimen. This result is in agreement with that of [36], who explained that the weight gain after early exposure to sodium sulphate solution (up to 90 days) could be due to the sulphate salt and hydration products were formed.

### **Strength loss**

Figure 9 presents the compressive strength loss of the RHA and CB mixtures at different curing ages before exposure to the sodium sulphate solution. The overall trend shows a decrease in the strength loss with prolonged curing age for all mixtures.

As for the RHA specimens (Figure 9(a)), it is constantly observed that the strength loss gradually diminishes as the RHA replacement ratio increases. For example, the loss in compressive strength of the 56-day control specimen was 1.15% while the strength loss of the 56day specimens incorporating 10%, 20%, and 30% RHA content was found to be 0.85%, 0.55%, and 0.32%, respectively. Furthermore, the strength loss of the 56day specimen containing 10% CB content was 0.98% in which was lower than that of the control specimen by 14.78% (Figure 9(b)). However, an increase in the CB replacement ratio up to 30% slightly extended the strength loss by 34.69% compared to that of specimen containing 10% CB content.



Figure 7: Strength loss of POBC mortar containing RHA (a) and CB (b) after exposure to sulphuric acid solution



Figure 8: Weight gain of POBC mortar containing RHA (a) and CB (b) after exposure to sodium sulphate solution

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Figure 9: Strength loss of POBC mortar containing RHA (a) and CB (b) after exposure to sodium sulphate solution

### Microstructural analysis

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The scanning electron micrographs of the control specimen, specimen incorporating 10% RHA, and 10% CB at the ages of 56 days (before exposure to sulphuric acid and sodium sulphate solutions) are demonstrated in Figure 10. It can be observed that the microstructure of each POBC mortar mixtures consisted of 3 components, including POBC fine aggregate fragment, cement matrix, and interfacial transition zone (ITZ) located between the POBC fine aggregate and the cement matrix. As depicted in Figure 10(a)-10(c), the POBC fine aggregate had an angular shape and smooth surface with subconchoidal fracture. In addition, the mixture containing RHA also provided a denser ITZ structure when compared to those of control specimen and CB specimen. The denser ITZ could contributed to the compressive strength improvement of the POBC mortar.

After 42 days of exposure to the sulphuric acid solution (pH≈2.5), alterations of the hydration products were observed under the scanning electron microscope. The photomicrographs of 56-day OPC specimen, specimens incorporating 10% RHA, and 10% CB are presented in Figure 11. It can be seen that the acid attack promoted some disintegration features within the microstructures, which accounted for both a weight loss and a strength reduction.



Figure 10: Scanning electron micrographs of the 56-day OPC specimen (a), specimen incorporating 10% RHA (b), and 10% CB (c) before exposure to sulphuric acid and sodium sulphate solutions







Figure 11: Scanning electron micrographs of the 56-day OPC specimen (a), specimen incorporating 10% RHA (b), and 10% CB (c) after exposure to sulphuric acid solution for 42 days

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The deleterious mechanism of acid attack is the dissolution of calcium hydroxide (a hydration product of cement paste) and the formation of gypsum (Ca<sub>3</sub>SO, 2H<sub>3</sub>O) and ettringite (Ca<sub>6</sub>Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>(OH)<sub>12</sub>.26H<sub>2</sub>O) [37-38]. As a result, the softening of cement paste and the propagating of some microcracks could lead to the weakening of the mortar specimen. The gypsum (a light monoclinic shape with one direction cleavage) and the ettringite (needle-like or elongate prismatic shape) crystals embedded in the matrix of the control specimen, as depicted in Figure 11(a). While a small amount of gypsum and ettringite crystals were observed in the specimens incorporating the RHA and CB supplements, as shown in Figure 11(b) and 11(c). In addition, the control specimen revealed less compactness, high porosity, and loosed matrix. Contrastingly, the specimens incorporating either the RHA or CB supplements displayed a denser matrix occupied by the C-S-H framework.

The SEM micrographs of the 56-day OPC specimen, specimen incorporating 10% RHA, and 10% CB exposed to sodium sulphate solution (pH=8) for 42 days, are illustrated in Figure 12. It is apparent that the specimens incorporating the RHA and CB exhibited lesser pore space and denser matrix (clogged up by C-S-H), compared to that of the control specimen. Moreover, the less ettringite and microcracks were observed in the specimens incorporating the RHA and CB supplements, as shown in Figure 12(b) and 12(c), when compared to that of the control specimen (Figure 12(a)).

In general, the external sodium sulphate attack mechanism is the penetration of dissoluble sulphate salt into the cement matrix which resulted in weight gain at the early sulphate exposure [39]. Such an attack can provide the deleterious effects on the cement matrix by inducing in the precipitation of sulfoaluminate phase ettringite (E) and gypsum (G) due to the destabilized calcium hvdroxide in cement paste reacts with dissoluble sulphate salt. Additionally, the formation of ettringite causes an increase in the volume of the cementitious matrix and induces some micro cracks. Subsequently, the expansion and microcracks could lead to the strength reduction [40-41]. An increase in the densification of the matrix within the POBC mortar incorporating the RHA and CB supplements could contributed to the pore refinements

and reduction of the connectivity of pores, which in turn mitigates the deterioration from the penetration of the dissoluble sulphate salt into the matrix.

### **CONCLUSIONS**

This study investigated the performance of mortar containing waste materials namely, palm oil boiler clinker as a fine aggregate substitution, the rice husk ash and calcium bentonite as a partial cement replacement. The compressive strength, water absorption, porosity, durability against sulphuric acid and sodium sulphate attacks at the age of 7, 28, and 56 days were examined. Based on the experimental results, the following conclusions are drawn:

- The compressive strengths of the POBC mortar incorporating the rice husk ash (RHA) and calcium bentonite (CB) were enhanced by a prolonged period of curing up to 28 days. The highest SAI of the RHA and CB mixtures at the age of 28 and 56 days was obtained in the mixtures containing RHA content of up to 20% and CB content of 10%.
- The increase in the RHA replacement ratio and the curing ages of POBC mortar have a positive impact on reducing both the water absorption and porosity. Additionally, the POBC mortar incorporating up to 10% CB provided lower water absorption and porosity compared to the control mixture. However, any increase in the CB replacement ratio of more than 10% significantly increases the water absorption and porosity
- The weight variation and strength loss due to sulphuric acid and sodium sulphate attacks of each POBC mortar mixtures were mitigated by increasing the curing duration before such exposure. Evidently, the POBC mortars incorporating the RHA supplement of up to 30%, and 10% of CB yielded the optimum durability against sulphuric acid and sodium sulphate attack.
- The SEM micrographs revealed that the interfacial transition zones were found to be denser in the mixture incorporating RHA. After exposure to sulphuric acid and the sodium sulphate solutions, the POBC mortars incorporating the RHA and CB provided less







Figure 12: Scanning electron micrographs of the 56-day OPC specimen (a), specimen incorporating 10% RHA (b), and 10% CB (c) after exposure to sodium sulphate solution for 42 days

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disintegration combined with a dense matrix occupied by C-S-H.

The POBC mortar in this study can be served as the non-heavy duty construction materials, eco-brick, and eco-mortar block productions. Besides, the supplement of up to 30% RHA and 10% CB can be possibly added as an OPC replacement to alleviate the cement consumption and promote the performance of the POBC mortar against the non-aggressive environment.

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### **APPENDIX D**

## **Research article 2(Indexed by TCI, tier 1)**

Kueaket, K., & Tonnayopas, D. (2021). Sustainable mortar and concrete made from palm oil boiler clinker aggregate comprising rice husk ash and calcium bentonite: Compressive strength and durability assessment. *Journal of Applied Science*, 20(1), 39-55. doi: 10.14416/j.appsci.2021.01.004

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**Research Article** 

### Sustainable mortar and concrete made from palm oil boiler clinker aggregate comprising rice husk ash and calcium bentonite: Compressive strength and durability assessment

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### **Abstract**

This research was conducted to assess the performances of mortars and concretes made from palm oil boiler clinker aggregate (POBC) blending with rice husk ash (RHA) and calcium bentonite (CB). These blends were used to substitute of ordinary Portland cement (OPC) in the combined proportions of 5wt%CB+5wt%RHA, 5wt%CB+15wt%RHA, 15wt%CB+5wt%RHA, 10wt%CB+20wt%RHA, and 20wt%B+10wt%RHA. The POBC mortars were examined for compressive strength, water absorption, sulfuric acid resistance, and microstructural characterization at the age of 7, 28, and 56 days. Subsequently, the POBC concretes were tested for rapid chloride ion permeability and capillary absorption at 28 and 56 days of curing ages. As a result, the 56-day POBC mortar with the optimum blending of 5% CB and 15% RHA provided the greatest compressive strength of 48 MPa as well as revealed denser microstructure observed under scanning electron microscope (SEM) and greater durability against sulfuric acid attack which surpassed those of the POBC mortar without CB and RHA. Proportionately, the incorporation of 5-10% CB with 15-20% RHA to POBC concrete cured up to 56 days possessed greater durability performances by reducing the chloride ion permeability from moderate to very low ranges as well as lessening capillary absorption. The practical implications of this study are that the POBC could be potentially used in the building sector as a substitute to the conventional aggregates. In addition, the use of CB and RHA could be possibly replaced cement in the optimum proportions without compromising the strength and durability to produce efficiently sustainable POBC mortar and concrete.

Keywords: palm oil boiler clinker, rice husk ash, calcium bentonite, compressive strength, sulfuric acid resistance, chloride ion permeability

### **Introduction**

In 2021 to 2022, the prediction of construction aggregate demand in Asia-Pacific region would be reached to approximately 45,000 million of tonnes (Wiwattananukul et al., 2019). Specifically, the estimate construction aggregate demand in Thailand was 50 million of tonnes annually, and it was predicted to be increased by 20% of each year (Material resources management division, 2019). Conversely, the construction aggregate reserve was approximately 8,010 million of tonnes in Thailand (Innovation in Raw Materials and Primary Industries Division, 2020). Thus, it is apparent that the construction aggregate demand has been inversely

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proportional to the nonrenewable aggregate reserve. Therefore, the alternative construction aggregate sources should be essentially studied and further brought to use. The demolition waste of construction and industrial waste have been widely applied in the sustainable construction material sectors.

Utilization of local waste materials in construction applications has a great impact on reducing the waste itself, land-fill used, environmental pollutions, and consumption of natural construction materials (Halahla et al., 2019). These impacts also promote sustainable construction towards waste manipulation. To reduce the consumption of the natural construction material and increase the exploitations of waste by-products, the natural aggregate and the cement can be efficiently replaced by the waste by-products such as copper slag (Siddique et al., 2020), granulated blast-furnace slag (Maghool et al., 2020), brick waste (Dang & Zhao, 2019), and fly ash (Chindaprasirt et al., 2020; Klimek et al., 2020; Shakir et al., 2020; Siddique et al., 2020).

Palm oil boiler clinker (POBC) was considered as a rigid solid waste which obtained from local crude palm oil plant. This waste was generated after the combustion of solid palm oil wastes inside boiler and caused the disposal area used and environmental pollutants (Hamada et al., 2020). In regard to the rigid properties and chemical compositions of POBC, the feasibility usages had been studied in term of sustainable construction application as a building material replacements (Shakir et al., 2019). According to Chai et al (2017), POBC can be potentially used as up to 50% of coarse aggregate substitution in size of 4.75-9 mm. Besides, at 100% of POBC coarse aggregate replacement for concrete with w/b ratio of 0.53, the 28-day compressive strength was up to 33 MPa which was only 30% lower than the reference mix made of granite aggregate (Abutaha et al., 2016). Moreover, natural sand can be completely substituted by POBC fine aggregate for eco-mortar production with a minor effect on compressive strength (Kanadasan et al., 2018). The percentage replacement of natural sand by POBC was recommended between 40 to 60% to maintain the mechanical properties (Babalghaith et al., 2020). Although, the studies of using POBC as an aggregate substitution in concrete, asphalt, and geopolymer productions and its mechanical properties have been reported by several researchers, the durability of those composites have not yet been clearly characterized in order to determine the suitability usage and application of POBC. Besides, the study of using 100% of POBC as coarse and fine aggregate with other type of waste as a binder and its durability characteristics have not been reported elsewhere.

Additionally, rice husk ash (RHA) is one of the most useful materials in cement industry. Several important roles of RHA as a pozzolan in cementitious material had been revealed, it could reduce the capillary pore and promote long term compressive strength of concrete, especially, for the low carbon RHA (Kang et al., 2019; Sandhu & Siddique, 2017). The pozzolanic activity was promoted by the addition of RHA which resulted in the micro-filler effect that filled up the capillary pore of cement matrix by the production of calcium silicate hydrate gels (C-S-H gels) (Gill & Siddique, 2018). Besides, the efficiency of using RHA as a cement replacement on acid resistance was also found that concrete incorporating RHA performed the superior resistance due to the pozzolanic activity consumed calcium hydroxide (CH) content and strengthened the bond between different phases in microstructure by producing more C-S-H (Koushkbaghi et al., 2019). Conversely, the large amount of RHA replacement in concrete can be decreased the compressive strength (Umasabor & Okovido, 2018).

Furthermore, calcium bentonite (CB) was addressed for a natural pozzolan (Memon et al., 2012). The Pakistani bentonite or CB was partially replaced by weight of cement and found that the compressive strength of 56-day mortar containing 12-15% CB as a cement replacement can reach approximately 30 MPa, which was significantly 2.7% higher than that of without adding

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CB. The pore filling effect of using up to 10% CB replaced by weight of cement was achieved in mortar cured up to 28 days. This effect established the particle packing improvement and a decrease in water absorption due to the very-fine grain particles of CB (Ahmad et al., 2011).

This research aims to utilize the local waste by-products and evaluate the performances of mortars and concretes containing POBC aggregate with the combination of RHA and CB as a cement replacements. The compressive strength, water absorption, and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) resistance of POBC mortars were carried out at the age of 7, 28, and 56 days. In addition, the POBC concretes cured at 28 and 56 days were determined for rapid chloride ion permeability and 72-hour capillary absorption. Accordingly, the microstructures of POBC mortar containing CB and RHA blends were characterized via scanning electron microscope (SEM).

### **Materials and methods**

Ordinary Portland cement (OPC) as per ASTM C150 was used in this study. POBC was collected from disposal area of oil palm plant at Surat Thani province, as received POBC was displayed in Figure 1a. The POBC was washed, dried, and crushed with jaw crusher to achieve the POBC coarse aggregate (POBCCA) and gyratory crusher to achieve the POBC fine aggregate (POBCFA), as depicted in Figure 1b-c. The enlargement of the POBC mortar's cross-section obtained from stereo microscope is shown in Figure 1d. The mineralogical characteristics of POBC were characterized via X-ray diffraction (XRD) analysis, as shown in Figure 2. The mineral compositions of POBC consisted of whitlockite, diopside, and three polymorphs of SiO2, which were quartz, tridymite, and crystobalite. The green color of POBC can be accounted for the mineral composition of diopside. The properties of POBC aggregates were listed in Table 1. Conforming to ASTM C131 (2020) and BS 182-112 (1990), the POBCCA is not recommended for using in road surfacing, structural works, or heavy duty constructions due to the lower resistance to crushing and abrasion. In addition, CB was purchased from supplier (product of Thailand), as seen in Figure 1b.

RHA was obtained after the open air burning of using rice husk as a biomass fuel in Phatthalung province. Then, it was calcined with an electrical furnace at 700 °C for 1 hour (HR: 5 °C/min) in order to reduce the contaminated organic matters or unburned carbon. After calcining process, RHA was ground with jar mill at rotation speed of 65 revolution per minute for 12 hours to meet the requirement of ASTM C618 (2019). The chemical compositions and particle size of CB and RHA were analyzed via wavelength dispersive X-ray fluorescence spectrophotometer (WDXRF) and laser particle size analyzer (LPSA), as tabulated in Table 2. The XRD pattern of RHA is depicted in Figure 3. The RHA composed of few crystalline phase of crystobalite and quartz, mostly contained amorphous silica, which confirmed by the broad hump peak between 20 of 15° to 25° and other wide range of low intensity peaks, as presented in the dashed line circle. The particle size distributions of the binders and aggregates used are shown in Figure 4.

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**Figure 1** As received POBC (a), from left to right: crushed POBC as a fine aggregate, rice husk ash, and calcium bentonite (b), enlargement of POBC fine aggregate (c), and cross-section of mortar made of POBC fine aggreg



Figure 2 XRD pattern and phase compositions of POBC



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Table 1 The physical properties of POBC aggregates









Figure 3 XRD pattern and phase compositions of RHA



Figure 4 Particle size distribution of POBC aggregates and binders

### **Mix designation**

Mix designations of POBC mortar and concrete were done complying with ASTM C109 (2016), ASTM C192 (2019), and ACI 211.1 (1991). For both of POBC mortar and concrete, the blending of RHA and CB partially replaced by weight of OPC, as followed in Table 3. For example, the mix designation B5R5 was the combination between 5% CB and 5% RHA replaced by weight of OPC. The POBC mortar specimen was taken for determine the compressive strength, water absorption, and resistance to sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) attack. The POBC concrete specimen was taken for determine the rapid chloride permeability and capillary absorption. For the POBC mortar mix designations, 50-mm cube specimens were casted with the binder to aggregate ratio of 1:2.75 and water to binder ratio of 0.48. The percentage flow of fresh mortar was determined as per ASTM C1437 (2015). Then, the hardened mortar specimens were cured in saturated lime water at ambient temperature (25±5°C) with 80-85% relative humidity for 7, 28, and 56 days. For the POBC concrete mix designations, 100 mm diameter with 50 mm length concrete discs were prepared by using natural sand (F.M 3.15), POBCCA with maximum size of 10 mm (F.M. 5.31), and taken water to binder ratio of 0.48. The concrete specimens were cured in the same condition of POBC mortar for 28 and 56 days.

Table 3 The mix designations of POBC mortar for various combination ratios of RHA and CB



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### **Test procedures**

The compressive strength and water absorption of POBC mortar were prepared and tested in accordance with ASTM C109 (2019) and ASTM C373 (2014). The strength activity index was determined according to ASTM C618 (2019). Resistance to 0.005 M sulfuric acid attack (pH=2.5) was measured by the comparison between the weight and compressive strength of POBC mortars before exposure and after exposure to sulfuric acid solution at particular curing ages. The sulfuric acid was renewed every week in order to maintain the pH value of the solution. The sulfuric acid resistance test was conducted for 6 weeks. The rapid chloride permeability test and capillary absorption test of POBC concretes were performed conforming to ASTM C1202 (2019) and ASTM C1585 (2020). The tests set up of the rapid chloride ion permeability and the capillary absorption are presented in Figure 5. For the microstructural characterization, the POBC mortars cured at 56 days were investigated using scanning electron microscope (SEM) at the accelerate voltage of 20 kV.



Figure 5 Schematics of rapid chloride permeability (a) and capillary absorption test (b) configurations

### **Results and discussion** 1. Compressive strength

The compressive strength of mortar specimens cured in saturated lime water for a period of 7, 28, and 56 days are shown in Figure 6. Overall, the compressive strength of all mix designations was increased with curing ages. It can be observed that the compressive strength of mix designations B10R20 and B5R15 were equivalent to or higher than that of the control mix at the age of 28 and 56 days. Particularly, the highest 56-day compressive strength of 48 MPa was found in the mix B5R15, which was greater than that of the control mix by 7%. However, a reduction in 56-day compressive strength was found in the mix B5R5, B15R5, and B20R10. Which was lesser than that of the control mix by 11%, 16%, and 15%, respectively. According to these results, it can be seen that the mix designations containing more RHA/CB ratios (B5R15 and B10R20) and taken up to 28-day curing ages could be maintained and provided the development of the compressive strength in comparison to the control mix, B5R5, B15R5, and B20R10. These compressive strength results comply with those of (Joshaghani & Moeini, 2018). Who reported that the compressive strength of 90-day mortar containing 15% RHA was 6% greater than that of the mixture without RHA. Additionally, the compressive strength of 28-day mortar specimen containing 6% bentonite was higher than that of the control specimen by 3% (Memon et al., 2012). However, the effects of hybrid mixes between RHA and CB have not been studied and reported elsewhere. Therefore, according to the compressive strength results of this study, the mix designation B5R15 and B10R20 can be recommended as the optimum CB/RHA ratios for using as an OPC replacement without detrimental to the compressive strength.

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Figure 6 Compressive strengths of POBC mortars with different mix designations at the age of 7, 28, 56 days

### 2. Strength activity index

The strength activity index (SAI) of all mix designations is demonstrated in Figure 7. The determination of SAI is to indicate that the SCMs or any tested materials have the pozzolanic properties. According to ASTM C618 (2019), the requirement of SAI is considered as more than 75%. For the mixture containing the hybrid mixes of RHA and CB, the SAI values of those mix designations were in the range of 76-111%. In addition, the SAI of those mixes were meet the standard requirement and significantly increased with ages. In addition, the highest SAI was found in the mix B5R15 at the age of 28 days. This finding is in line with Man et al. (2019) and Rehman et al. (2020). The development of SAI and compressive strength could be due to the chemical compositions of RHA and CB. Which mainly consisted of silica (SiO<sub>2</sub>). Moreover, the amount of  $SiO_2$ + Fe<sub>2</sub>O<sub>3</sub>+ Al<sub>2</sub>O<sub>3</sub> contents were more than 70%. Thus, the active silica could be reacted with Ca(OH)<sub>2</sub> and promote and pozzolanic reaction's products caused the improvement in compressive strength (Man et al., 2019; Rehman et al., 2020).



Figure 7 Strength activity index of POBC mortars with different mix designations at the age of 7, 28, 56 days

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### 3. Water absorption

The water absorption of different mix designations at the ages of 7, 28, and 56 days is shown in Figure 8. The water absorption of all mix designations was decreased with prolonged curing ages. Meanwhile, at the early age of curing, the water absorption of the hybrid mix designations was similar to the control mix. At the age of 28 and 56 days, the percentage of water absorption was decreased by 15 to 26% for the mix B5R5, B5R15, and B10R20, when compared to that of the control mix. The lowest percentage of water absorption was found in the mix B5R15 at the age of 28 and 56 days, which was 26% lesser than the control mix. Jamil et al. (2016) pointed out that the filler effect and secondary C-S-H products from pozzolanic reaction of RHA filled up the pore space in the cement matrix and caused the reduction in the water absorption. Furthermore, the reduction in the water absorption was also found in the specimen containing up to 10% bentonite. This is due to the micro-filling effect of very fine-grain bentonite that can improve the particle packing of binder and reduce the water to binder ratio (Ahmad et al., 2011; Mesboua et al., 2018). However, an adverse effect of more bentonite contents could be attributed to the unreacted bentonite particles within the cement matrix absorbed more water, thus the water absorption of specimen containing more bentonite increased (Ahmad et al., 2011).

Although, the control mix revealed the highest 56-day water absorption while provided the greater compressive strength than the mix designation B5R5, B15R5, and B20R10. This could be due to the complex mechanism between RHA and CB, which contributed greatly to both of the pozzolanic reaction and filler effect. For the mechanism of RHA, the very-fine grain particle substantially improved pore size, reduced water absorption, and was ineffective to the strength development due to the pozzolanic reaction takes place in prolonged curing period. Moreover, this mechanism was confirmed by Bheel et al. (2020) that an increase in dosage of RHA replacement decreased the water absorption, whereas only the optimized 5% of RHA increased the compressive strength at the age of 28 days. Correspondingly, the significance mechanism of CB is pore refinement and improving the impermeability of cement matrix (Masood et al., 2020; Rehman et al., 2019). Liu et al. (2020) also concluded that the incorporating 10% CB developed the durability by filling effect and only the C-S-H gel formed on the surface of the specimen, whereas the strength development index need to be included (i) the forming of ettringite and (ii) the overlapping of hydration product on the specimen's surface.



Figure 8 Water absorption of POBC mortars with different mix designations at the age of 7, 28, 56 days

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### 4. Resistance to sulfuric acid attack **Weight loss**

The weight loss of mortars at different curing ages exposed to 0.005 M sulfuric acid solution for 42 days is shown in Figure 9. It can be observed that the weight loss of all mix designations was decreased with curing age before exposure to sulfuric acid solution. At the later age, 56 days of curing, the ranges of weight loss for all hybrid mix designations were lesser than the control mix. Which were in the range of 1 to 44%. Especially, for the mix containing more combination ratio of RHA/CB (B10R20 and B5R15), the weight loss was up to 41 and 44% lower than that of the control mix. This is mainly attributed to (i) the less water absorption and (ii) the pozzolanic activity from the silica compositions of RHA (94% SiO<sub>2</sub>) was greater than CB (57% SiO<sub>2</sub>) which contributed to an increase in C-S-H forming and resulted in more impermeable matrix. Thus, the more durability was obtained in the mix containing more RHA contents (Ahmad et al., 2011). Correspondingly, the mix designation B5R15 showed a lesser surface erosion than that of the control mix, as shown in Figure 10. Min & Song (2018) also reported that the crucial corrosion can be indicated in the yellow color area that appeared on the exposed surface of the cement composite to sulfuric acid solution.

### **Compressive strength loss**

The compressive strength loss of mortars exposed to 0.005 M sulfuric acid solution is shown in Figure 11. After 42 days of exposure, the compressive strength loss is seen in all mix designations. In accordance with the weight loss results, a similar trend was observed in the compressive strength loss. An increase in curing ages before sulfuric acid attack and the more RHA/CB ratios decreased the loss in compressive strength. The lowest compressive strength loss was found in the mix B5R15 at the age of 56 days. Which was 18 % lower than that of the control mix. This finding suggests that up to 20% of OPC can be replaced by the combination blend of CB and RHA at the optimum CB/RHA ratio as in the mix designation B5R15, which produced the development both in the compressive strength and durability.





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Figure 10 Enlargement of the surface of 56-day mortars: control mix (a) and mix designation B5R15 (b) after exposed to 0.005 M sulfuric acid solution for 42 days



Figure 11 Compressive strength loss of POBC mortars with different mix designations cured at 7, 28, and 56 days exposed to 0.005 M sulfuric acid solution for 42 days

### 5. Rapid chloride permeability and capillary absorption

The total charge passed and capillary absorption of different mix designations and curing ages are given in Figure 12. According to ASTM C1202 (2019), the qualitative indication of chloride ion permeability of any concrete specimen was determined as followed: the total charges passed of concrete specimens that fall in the range of (i) >4,000 coulombs, (ii) 2,000-4,000 coulombs, (iii) 1,000-2,000 coulombs, and (iv) 100-1,000 coulombs were considered as high permeability, moderate permeability, low permeability, and very low permeability, respectively. A decrease in chloride ion permeability was observed in all hybrid mixes with prolonged curing ages. As compared to the control mix, the decrease was in the range of 4-77% and 30-78% at the age of 28 and 56 days, respectively. At the age of 56 days, the chloride ion permeability of the mix designations B5R15 and B10R20 was in very low ranges. For the mix designations B5R5, B15R5, and B20R10 cured at 56 days, the chloride ion permeability was in the low range, whereas the chloride ion permeability of the control mix was in the moderate range. Consequently, the capillary absorption results corresponded to the chloride ion permeability. An increase in curing ages decrease capillary absorption. The significant reduction in the 72-hour capillary absorption

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was found in the mix designations B5R15 and B10R20 at the age of 56 days, which were lower than the control mix by 44% and 56%, respectively. It can be seen that the chloride permeability and the capillary absorption decreased with an increase in curing ages and RHA/CB ratios, as was found in mixture designations B5R15 and B10R20. Ganesan et al. (2008) also found that the chloride permeability and rate of capillary absorption had a good relationship due to the reduction of permeable voids in matrix by the pozzolanic activity of RHA as an additive and the prolonged curing ages. This finding also in agreement with those of Huang et al. (2017), Laidani et al. (2020), and Zareei et al. (2017) that the capillary absorption and chloride ion penetration of concrete specimen containing RHA were reduced by an increase in the RHA content, while only adding up to 10% bentonite could reduce the capillary absorption. Moreover, an increase in RHA content up to 20% reduced the 72-hour capillary absorption by 15%, compared to the specimen without RHA (Zahedi et al., 2015).



Figure 12 The chloride ion permeability and 72-hour capillary absorption of POBC concrete with different mix designations at the age of 28 and 56 days

### 6. Microstructural characterization

The SEM images are demonstrated the comparison between OPC particles and combination blends of OPC, RHA, and CB particles of the mix designation B5R15 as shown in Figure 13a-b. It can be seen that the different in particle sizes among OPC, RHA, and CB played the important role in increasing the particle packing of the binder. An increase in particle packing was observed in the binder of the mix designation B5R15 in comparison to the pure OPC particles. Accordingly, the very fine micro-particles of CB and rounded agglomerate RHA particles also contributed to the reduction in water content of the binder due to RHA and CB absorbed more water (Ahmad et al., 2011; Mesboua et al., 2018). The SEM images of the hardened pastes of the control mix and mix designation B5R15 are illustrated in Figure 13c-d. It can be clearly seen that the hydration's products were found in both mix designations. Particularly, the mix designation B5R15 manifested denser matrix and lower micro-pore spaces when compared to the control mix. The dense flocs-like C-S-H was obviously found in the groundmass of the mix

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designation B5R15 and the micro-pore spaces were reduced by the spreading of the dense C-S-H framework. For the matrix of the control mix, the loosely fibrous-like and flocs-like C-S-H were found. The more C-S-H embedded in the mix containing RHA and CB could be due to the pozzolanic activity that consumed free Ca(OH)2 from both of hydration and pozzolanic reaction's products, then produced more C-S-H (Chopra et al., 2015; Masood et al., 2020). Besides, the denser microstructure could be attributed to the development in compressive strength, the reduction in water absorption, and the durability improvement of the mix designation B5R15 in comparison to the control mix. The morphology of C-S-H observed from SEM images of this study was characterized and compared to the flocs-like C-S-H found by Yu et al. (1999), Cizer et al. (2007), and the fibrous-like C-S-H found by El-gama et al. (2018). However, the characteristics of C-S-H can be shown in various forms depended on the curing condition, additive used, water to binder ratios, and designation of mixture (Cizer et al., 2007; Shen et al., 2019).



Figure 13 SEM images of OPC particles (a), blends consisted of OPC, CB, and RHA particles of mix designation B5R15 (b), the hardened paste cured at 56 days of the control mix (c), and the mix designation B5R15 (d)

### **Conclusion**

Based on this study results, the conclusions can be provided as following:

(1) The POBC mortar with the optimum combination of 5% of CB and 15% of RHA replaced by weight of OPC (mix designation B5R15) developed the superior performance at the curing age up to 56 days when compared to the control and the others mix designations. Moreover, the mix designation B5R15 provided the highest compressive strength, the lowest water absorption, and the lowest weight and compressive strength loss due to sulfuric acid attack.

(2) The chloride ion permeability and capillary absorption decreased with an increase in RHA combination ratios and curing ages. The very low range of chloride ion permeability belonged

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to the mix designation B5R15 and B10R20 cured at 56 days. However, the chloride ion permeability of the others mix designations were fallen into the low range except for the control mix that was continually fallen in the moderate range when the curing ages increased. The optimum combination of 5-10% of CB and 15-20% of RHA replaced by weight of OPC would be recommended for the chloride ion permeability resistance of the POBC concrete.

(3) According to the microstructure characterization using SEM images, the denser matrix occupied by C-S-H framework and lower pore spaces of the mix designation B5R15 in comparison to the control mix contributed to the compressive strength and durability improvement.

(4) Regarding to this investigation, the POBC can be used as an aggregate substitution for producing conventional mortars and concretes in non-structural construction work. The optimum combinations of CB and RHA for POBC mortars and concretes could help diminishing the cement used and promoting the durability performance.

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## **APPENDIX E**

Experimental materials preparation and mix designation

### **Experimental materials preparation and mix designation**

### **1. Preparing POBC aggregates**

The POBC raw samples were washed with tap water, scrubbed with brush and towel to diminish access clay minerals and organic matters from the waste disposal areas, then sun drying. After that, the POBC will be break into smaller size by sledge Hammer, crushed with jaw crusher for coarse aggregate and gyratory crusher for fine aggregate. The preparation process of POBC aggregate is illustrated in Figure 37.



Figure 37 Flow chart of preparing POBC aggregate

### **2. Preparing RHA**

The RHA obtained from the open-air burning of the rice husk for 72-84 hours until the RHA was cooled down. Then, the RHA was incinerated at 700 ºC by an electrical furnace and ground with jar mill for 12 hours (60-65 rpm). After that, the RHA was sampling and tested for laser particle size analysis. According to the test results (see in the results and discussions section), the particle size of RHA was lesser than 45 microns. The preparation process of RHA is presented in Figure 38.



Figure 38 Flow chart of preparing RHA
#### **3. Mix designation**

The mixtures were designed in 3 levels for an OPC replacement. The RHA and CB were partially replaced an OPC, type 1 by weight of 10%, 20%, and 30%. The mixtures named R10, R20, and R30 were the mixes containing RHA as an OPC replacement for 10%, 20%, and 30%, respectively. The binary mixtures named B10, B20, and B30 were the mixes containing CB as an OPC replacement for 10%, 20%, and 30%, respectively. The ternary mixtures of CB and RHA named B5R5, B5R15, B15R5, B10R20, and B20R10. The proportions of these mixes for mortars and concretes were tabulated in Table 13. The mortar and concrete specimen will be cured in saturated-lime water at 29±3 ºC for 7, 28, 56 days and 28, 56 days, respectively. The mortar was designed as in accordance with ASTM C109 (2020). In addition, the concrete mix designs were based on ACI 211.1 (1991) for achieve compressive strength at 35 MPa in 28 days. The mortar and concrete specimens are shown in Figure 39.

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			Mix proportion (kg/m <sup>3</sup> )						
No.	Mix designation	w/b ratio	<b>OPC</b>	Sand	<b>POBCA</b>	<b>POBCFA</b>	CB	<b>RHA</b>	
				POBC mortar					
1	Control	0.48	500			1,375			
$\overline{2}$	R <sub>10</sub>	0.48	450			1,375		50	
3	R <sub>20</sub>	0.48	400	-		1,375		100	
4	R30	0.48	350			1,375		150	
5	<b>B10</b>	0.48	450			1,375	50		
6	<b>B20</b>	0.48	400			1,375	100		
$\overline{7}$	<b>B30</b>	0.48	350			1,375	150		
8	<b>B5R5</b>	0.48	450			1,375	25	25	
9	<b>B5R15</b>	0.48	400			1,375	25	75	
10	<b>B15R5</b>	0.48	400			1,375	75	25	
11	<b>B10R20</b>	0.48	350			1,375	50	100	
12	<b>B20R10</b>	0.48	350			1,375	100	50	

Table 14 Mix proportions of POBC mortars and concretes

	POBC concrete									
No.		w/b ratio	Mix proportion (kg/m <sup>3</sup> )							
	Mix designation		<b>OPC</b>	Sand	<b>POBCA</b>	<b>POBCFA</b>	CB	<b>RHA</b>		
13	Control	0.48	500	1,175	765					
14	R <sub>10</sub>	0.48	450	1,175	765			50		
15	R <sub>20</sub>	0.48	400	1,175	765			100		
16	R <sub>30</sub>	0.48	350	1,175	765		-	150		
17	<b>B10</b>	0.48	450	1,175	765		50			
18	<b>B20</b>	0.48	400	1,175	765		100			
19	<b>B30</b>	0.48	350	1,175	765		150			
20	<b>B5R5</b>	0.48	450	1,175	765		25	25		
21	<b>B5R15</b>	0.48	400	1,175	765		25	75		
22	<b>B15R5</b>	0.48	400	1,175	765		75	25		
23	<b>B10R20</b>	0.48	350	1,175	765		50	100		
24	<b>B20R10</b>	0.48	350	1,175	765		100	50		

Table 14 Mix proportions of POBC mortars and concretes (Continue)



Figure 39The POBC mortar specimens (A) and the POBC concrete prepared for RCPT test (B)

# **APPENDIX F**

Properties of POBC mortars



Table 15 Compressive strength, apparent porosity, and water absorption of POBC mortars with different curing ages



















Table 15 Compressive strength, apparent porosity, and water absorption of POBC mortars with different curing ages (Continue)



\*Oven-dry weight at 50 °C for 72 hours













\*Oven-dry weight at 50 °C for 72 hours









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#### **APPENDIX G**

Properties of POBC concretes

	Ages		28 days		56 days		
		$\mathbf{1}$	107.84		107.43		
	Diameter (mm)	$\overline{2}$		107.93	107.12		
		Average		107.89	107.28		
		$\pmb{0}$	0.140	0.140	0.130	0.130	
		$30\,$	0.170	0.340	0.130	0.260	
		60	0.180	0.360	0.140	0.280	
		90	0.180	0.360	0.140	0.280	
		120	0.185	0.370	0.150	0.300	
Control	Time elapsed (min)	150	0.190	0.380	0.150	0.300	
		180	0.190	0.380	0.160	0.320	
		210	0.190	0.380	0.170	0.340	
		240	0.190	0.380	0.180	0.360	
		270	0.190	0.380	0.180	0.360	
		300	0.190	0.380	0.190	0.380	
		330	0.190	0.380	0.190	0.380	
		360	0.190	0.190	0.190	0.190	
		$Q_x$ (Coulombs)	3,978		3,492		
		$Q_s$ (Coulombs)	3,085		2,739		

Table 18 The RCPT results of POBC concrete with different binary and ternary mixtures and curing ages

	Ages		28 days		56 days		
		$\mathbf{1}$		107.89	107.45		
	Diameter (mm)	$\overline{2}$		107.90	107.87		
		Average		107.90	107.66		
		$\mathbf 0$	0.060	0.060	0.045	0.045	
		30	0.060	0.120	0.045	0.090	
		60	0.060	0.120	0.045	0.090	
		90	0.070	0.140	0.045	0.090	
	Time elapsed (min)	120	0.070	0.140	0.045	0.090	
R10		150	0.070	0.140	0.045	0.090	
		180	0.070	0.140	0.045	0.090	
		210	0.070	0.140	0.045	0.090	
		240	0.080	0.160	0.045	0.090	
		270	0.080	0.160	0.045	0.090	
		300	0.080	0.160	0.045	0.090	
		330	0.100	0.200	0.045	0.090	
		360	0.100	0.100	0.045	0.045	
		$Q_x$ (Coulombs)		1,602	972		
		$Q_s$ (Coulombs)		1,242	757		

Table 18 The RCPT results of POBC concrete with different binary and ternary mixtures and curing ages (Continue)

	Ages			28 days	56 days			
		$\mathbf{1}$		107.23	107.87			
	Diameter (mm)	$\overline{2}$		107.46	107.80			
		Average		107.35	107.84			
		$\mathbf 0$	0.040	0.040	0.025	0.025		
		30	0.050	0.100	0.030	0.060		
		60	0.050	0.100	0.035	0.070		
		90	0.060	0.120	0.035	0.070		
	Time elapsed (min)	120	0.060	0.120	0.035	0.070		
<b>R20</b>		150	0.060	0.120	0.035	0.070		
		180	0.060	0.120	0.035	0.070		
		210	0.060	0.120	0.035	0.070		
		240	0.070	0.140	0.035	0.070		
		270	0.100	0.200	0.035	0.070		
		300	0.100	0.200	0.035	0.070		
		330	0.100	0.200	0.035	0.070		
		360	0.100	0.100	0.035	0.035		
		$Q_x$ (Coulombs)		1,512	738			
		$Q_s$ (Coulombs)		1,184		573		

Table 18 The RCPT results of POBC concrete with different binary and ternary mixtures and curing ages (Continue)









	Ages		28 days		56 days		
		$\mathbf{1}$	108.05		106.91		
	Diameter (mm)	$\overline{2}$		108.08	106.86		
		Average		108.07	106.89		
		$\mathbf 0$	0.010	2.000	0.080	0.080	
		30	0.130	0.130	0.100	0.200	
		60	0.140	0.280	0.100	0.200	
		90	0.150	0.300	0.100	0.200	
	Time elapsed (min)	120	0.170	0.340	0.100	0.200	
<b>B5R5</b>		150	0.180	0.360	0.120	0.240	
		180	0.185	0.370	0.120	0.240	
		210	0.190	0.380	0.120	0.240	
		240	0.190	0.380	0.120	0.240	
		270	0.190	0.380	0.120	0.240	
		300	0.190	0.380	0.120	0.240	
		330	0.190	0.380	0.120	0.240	
		360	0.190	0.380	0.120	0.120	
		$Q_x$ (Coulombs)		3,825	2,412		
		$Q_s$ (Coulombs)	2,956		1,905		

Table 18 The RCPT results of POBC concrete with different binary and ternary mixtures and curing ages (Continue)











	Ages		28 days		56 days		
		$\mathbf{1}$	106.86		107.27		
	Diameter (mm)	$\overline{2}$		107.62	107.52		
		Average		107.24	107.40		
		0	0.080	0.080	0.055	0.055	
		30	0.090	0.180	0.060	0.120	
		60	0.100	0.200	0.060	0.120	
		90	0.100	0.200	0.060	0.120	
	Time elapsed (min)	120	0.105	0.210	0.060	0.120	
<b>B20R10</b>		150	0.110	0.220	0.060	0.120	
		180	0.120	0.240	0.060	0.120	
		210	0.120	0.240	0.060	0.120	
		240	0.120	0.240	0.060	0.120	
		270	0.120	0.240	0.060	0.120	
		300	0.120	0.240	0.060	0.120	
		330	0.121	0.242	0.060	0.120	
		360	0.140	0.140	0.060	0.060	
		$Q_x$ (Coulombs)	2,405		1,292		
	$Q_s$ (Coulombs)			1,887		1,011	

Table 18 The RCPT results of POBC concrete with different binary and ternary mixtures and curing ages (Continue)

	Avg. Cross-sectional	Mass (kg)		$\Delta$ Mass/area*10 <sup>5</sup>	$\Delta$ Mass/area/time <sup>0.5</sup>	
Mix designation	area (mm <sup>2</sup> )	Weighted at 0 h	Weighted at 72 h			
Control	9,136.75	1.051	1.061	0.109	0.12	
R <sub>10</sub>	9,138.44	1.026	1.034	0.088	0.10	
R <sub>20</sub>	9,045.51	0.964	0.971	0.077	0.09	
R <sub>30</sub>	9,050.57	0.984	0.990	0.066	0.07	
<b>B10</b>	9,041.30	0.970	0.980	0.111	0.14	
<b>B20</b>	9,127.43	0.989	1.005	0.175	0.21	
<b>B30</b>	9,095.30	1.024	1.041	0.187	0.22	
<b>B5R5</b>	8,998.38	1.018	1.025	0.078	0.10	
<b>B5R15</b>	9,171.50	1.036	1.042	0.065	0.08	
<b>B15R5</b>	9,102.90	1.018	1.025	0.077	0.09	
<b>B10R20</b>	9,126.59	1.003	1.009	0.066	0.08	
<b>B20R10</b>	9,027.82	1.018	1.025	0.078	0.09	

Table 19 Capillary water absorption of 28-day POBC concrete with different binary and ternary mixtures

	Avg. Cross-sectional	Mass (kg)		$\Delta$ Mass/area*10 <sup>5</sup>	$\Delta$ Mass/area/time <sup>0.5</sup>	
Mix designation	area $(mm^2)$	Weighted at 0 h	Weighted at 72 h			
Control	9,007.64	1.039	1.047	0.096	0.10	
R <sub>10</sub>	9,139.29	1.041	1.047	0.075	0.08	
R <sub>20</sub>	9,122.36	1.018	1.023	0.064	0.07	
R30	9,118.97	1.019	1.024	0.056	0.06	
<b>B10</b>	9,058.16	1.004	1.013	0.103	0.11	
<b>B20</b>	8,945.52	1.086	1.095	0.108	0.12	
<b>B30</b>	8,969.00	1.014	1.027	0.151	0.17	
<b>B5R5</b>	8,963.96	1.055	1.062	0.079	0.09	
<b>B5R15</b>	9,061.53	1.042	1.048	0.067	0.07	
<b>B15R5</b>	8,958.93	1.031	1.037	0.078	0.08	
<b>B10R20</b>	8,994.19	1.003	1.008	0.066	0.07	
<b>B20R10</b>	9,075.03	1.036	1.042	0.071	0.08	

Table 20 Capillary water absorption of 56-day POBC concrete with different binary and ternary mixtures
<b>Mixes</b>	Diameter (mm)			L, Thickness (mm)						σt
	Top	<b>Bottom</b>	Avg.	Left	Right	Avg.	P(N)	2P(N)	$Pi^*(D)^*(L)$	(MPa)
Control	112.84	107.93	110.39	52.74	55.63	54.19	25,960	51,920	18,798.09	2.76
R <sub>10</sub>	107.89	107.90	107.90	51.43	50.82	51.13	32,180	64,360	17,336.41	3.71
R <sub>20</sub>	107.23	107.46	107.35	50.78	50.39	50.59	25,340	50,680	17,065.86	2.97
R <sub>30</sub>	107.33	107.42	107.38	51.35	52.20	51.78	22,190	44,380	17,472.21	2.54
<b>B10</b>	107.29	107.35	107.32	51.70	49.41	50.56	24,860	49,720	17,051.77	2.92
<b>B20</b>	107.94	107.72	107.83	51.90	50.79	51.35	24,110	48,220	17,400.53	2.77
<b>B30</b>	107.61	107.67	107.64	52.34	52.33	52.34	19,870	39,740	17,704.78	2.24
B <sub>5</sub> R <sub>5</sub>	108.05	108.08	108.07	50.79	50.75	50.77	24,400	48,800	17,243.16	2.83
<b>B5R15</b>	107.93	108.25	108.09	52.25	52.53	52.39	25,850	51,700	17,797.48	2.90
<b>B15R5</b>	107.70	107.61	107.66	51.25	51.82	51.54	24,710	49,420	17,436.57	2.83
<b>B10R20</b>	106.55	107.10	106.83	51.29	51.26	51.28	27,970	55,940	17,214.85	3.25
<b>B20R10</b>	106.86	107.62	107.24	51.76	52.02	51.89	24,590	49,180	17,489.01	2.81

Table 21 Indirect tensile strength of 28-day POBC concrete with different binary and ternary mixtures

<b>Mixes</b>	Diameter (mm)			L, Thickness (mm)						σt
	Top	<b>Bottom</b>	Avg.	Left	Right	Avg.	P(N)	2P(N)	$Pi^*(D)^*(L)$	(MPa)
Control	107.43	107.12	107.28	52.44	52.26	52.35	27,250	54,500	17,649.80	3.09
R <sub>10</sub>	107.45	107.90	107.68	51.11	52.57	51.84	33,350	66,700	17,543.03	3.80
R <sub>20</sub>	107.87	107.80	107.84	52.95	53.21	53.08	28,570	57,140	17,989.34	3.18
R <sub>30</sub>	107.74	107.78	107.76	52.65	51.35	52.00	25,840	51,680	17,611.06	2.93
<b>B10</b>	107.59	107.42	107.51	50.84	51.24	51.04	27,310	54,620	17,245.03	3.17
<b>B20</b>	106.69	106.75	106.72	52.66	51.59	52.13	26,310	52,620	17,483.02	3.01
<b>B30</b>	108.05	106.89	107.47	53.80	53.53	53.67	26,880	53,760	18,126.04	2.97
B <sub>5</sub> R <sub>5</sub>	106.90	106.86	106.88	51.58	52.34	51.96	26,560	53,120	17,453.81	3.04
<b>B5R15</b>	106.78	106.83	106.81	52.07	52.22	52.15	30,950	61,900	17,503.66	3.54
<b>B15R5</b>	107.85	107.44	107.65	52.05	52.59	52.32	27,010	54,020	17,700.53	3.05
<b>B10R20</b>	106.57	107.04	106.81	52.19	51.90	52.05	29,010	58,020	17,470.09	3.32
<b>B20R10</b>	107.27	107.52	107.40	53.57	52.20	52.89	26,810	53,620	17,850.12	3.00

Table 22 Indirect tensile strength of 56-day POBC concrete with different binary and ternary mixtures

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## **List of Publications and Proceedings**

- 1. Kueaket, K., & Tonnayopas, D. (2018). Enhanced properties of palm oil boiler clinker concrete with Sang Yod rice husk ash. *Proceeding of 7th International Conference on Applied Sciences and Engineering (ICASEA, 2018)*, Kota Kinabalu, Sabah, Malaysia, 29-38.
- 2. Kueaket, K., & Tonnayopas, D. (2019). Palm oil boiler clinker properties used as a green aggregate for construction. *Proceeding of the 13th International Conference on Mining, Materials, and Petroleum Engineering (CMMP 2019)*, Krabi, Thailand, 86-91.
- 3. Kueaket, K., & Tonnayopas, D. (2021). Compressive strength and durability performance of mortar containing palm oil boiler clinker aggregate, rice husk ash, and calcium bentonite. *Journal of Applied Engineering Science*, 19(1), 193-203. doi:10.5937/jaes0-27580
- 4. Kueaket, K., & Tonnayopas, D. (2021). Sustainable mortar and concrete made from palm oil boiler clinker aggregate comprising rice husk ash and calcium bentonite: Compressive strength and durability assessment. *Journal of Applied Science*, 20(1), 39-55. doi: 10.14416/j.appsci.2021.01.004