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Immobilization of Petroleum Sludge Incorporating Portland Cement and Rice Husk Ash

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Abstract—**Portland cement and rice husk ash were incorporated to stabilize and solidify the contaminant in petroleum sludge. Stabilization and solidification technique was chosen as an alternative treatment to reduce toxicity of the sludge prior to final disposal of the waste. The sludge has significant amount of organic material which normally interfere with the cement hydration process. A way to improve is by incorporation of cement replacement material. Mixture proportioning was conducted to find optimum water to cement ratio, sludge to cement ratio and cement replacement percentage. The solidified sludge performance was measured by compressive strength and permeable porosity. The optimum ratio of water to cement was found at 0.45 and cement to sludge of 8. Rice husk ash (RHA) was added at 5, 10 and 15 % cement replacement. 5 % RHA exhibited the best performance with regards to unconfined compressive of 24.9 N/mm^2. The strength was better than the sludge cement of 19.2 N/mm^2. Permeable porosity has inverse relationship with strength at water to cement ratio of 0.4. However at water to cement ratio of 0.45, the relationship showed different trend where increase in porosity cause increase in strength. Porosity was found to increase with increasing RHA content. The surface morphology of solidified cement with voids was found to be in the range of 10 to 15 µm for 15 % RHA.**

Index Terms—**compressive strength; permeable porosity; petroleum sludge; stabilization and solidification**

I. Introduction

Rapid economic growth and industrialization has contributed to generation of hazardous waste. Hazardous waste in nature has one or combination of the following properties, flammable, reactive, corrosive, ignitable and toxic. Potential health effects of the wastes, either acute or chronic are subjected to duration of exposure and dosage at the receiver. The toxic substances are classified according to the regulated bodies that control the discharges or deposit of waste from industrial sites. The toxic substances in the waste may migrate to the receiving stream and contaminate drinking water if not properly treated and disposed. Malaysian Department of Environment has introduced Scheduled Wastes regulation to control the disposal of toxic waste by adopting cradle to grave tracking system on 107 scheduled wastes. In 2008 more than a million metric ton scheduled wastes were produced in Malaysia [1].

Petroleum waste is one of the listed scheduled waste under Environmental Quality (Scheduled Waste) 2005 [2], and must be disposed off at a licensed landfill. Petroleum sludges are discharged from tank bottom, interceptors, wastewater treatment, biotreatment, contaminated soil, desalter, oil spill, filter clay, tar rags, and filter dust at maximum of 0.5 kg per ton of feedstock per refinery [3]. Massive waste generation involve high cost for thermal treatment.

Current practices to treat the waste included combustion and bioremediation. As an alternative, stabilization and solidification (S/S) technique should be considered as it had been practiced in western countries like U.S and Europe. Sludge disposal to soil is subjected to landfill regulation as in Australia and Norway; or waste management regulations in Malaysia, while Germany set threshold values for specific hazardous substance under water charges act.

II. Petroleum Sludge

A. Contaminants in Sludge

Petroleum sludge contains both inorganic and organic contaminants. Inorganic contaminants are in the form of metal compound such as zinc, lead, copper, nickel, chromium and mercury. Leaching of heavy metals into water intake points may cause health problems to human and aquatic organisms. Organic contaminants in the sludge measured as total petroleum hydrocarbon (TPH) are in the range of 510,000 to 640,000 mg/kg [4]. Only selected organic components receive attention due to its toxicity to human. Based on USEPA list, only 43 organic substances are more likely to be present and considered as potential environmental health concern in petroleum refining waste.

B. Treatment Objectives

The objective of the research is to reduce petroleum sludge toxicity using stabilization and solidification (S/S) based on ordinary Portland cement (OPC) as a main binder with selected additives for immobilization of contaminant in the sludge. Mixture proportioning was conducted to find the optimum cement to sludge ratio (C/Sd) and water to cement ratio (W/C) for stabilized and solidified sludge. The effect of cement replacement utilization on compressive strength was

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tested using rice husk ash (RHA) at 5 % to 15 % replacement. Permeable porosity of solidified products was investigated to find its relationship with strength.

C. Stabilization and Solidification

Stabilization reduces hazard potential of a waste by converting the contaminant into their least soluble, mobile or toxic state. Solidification refers to techniques that encapsulate the waste in a monolithic solid of high structural integrity [5]. Stabilization is a process of binder and additives used to reduce the solubility or chemical reactivity of toxic waste by changing its chemical state or by physical entrapment. Solidification attempt to convert the waste into easily handled solid with reduced hazards from volatilization, leaching or spillage [6]. The stabilization and solidification (S/S) are combined together and is termed "waste fixation."

D. Cement Reaction

Portland cement S/S is especially effective for waste with high level of toxic metals such as metal bearing wastes, activated sludges, fly ashes, lime/limestone wet scrubber, lead containing wastes, metal fines, inorganic sludge from electroplating and many others. S/S generally uses OPC Type 1 cement with additives such as fly ash, RHA and condensed silica fume.

Portland cement chemical reaction is exothermic with the heat of hydration evolves at the early hydration state as given in Equations 1 to 3. Main component of OPC are tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A) and tetracalcium aluminate ferric (C_4AF) forming calcium silicate hydrate (C-S-H), calcium hydroxide (CH), calcium sulfo-aluminate hydrate (ettringite, $C_6AS_3H_{32}$ or C_4ASH_{18}) and unhydrated clinker grain in solid forms [7]. Advantages of using Portland cement include low cost, long term stability, good impact and good compressive strength development [8]. Cement in combination with lime is sufficiently and economically viable for small industrial waste generators [9]. Portland cement and blended cement is used for study of leaching behaviour of alkanes and poly aromatic hydrocarbon in refinery sludge [10].

$$2C_3S + 6H \rightarrow C_3S_2H_3 + 3CH + 120\ cal / g. \qquad (1)$$

$$2C_2S + 4H \rightarrow C_3S_2H_3 + CH + 62\ cal / g. \qquad (2)$$

$$C_3A + CSH_2 \rightarrow Ettringite + 300\ cal / g. \qquad (3)$$

III. MATERIALS

A. Waste Material

Sludge sample collected from a refinery treatment plant was labeled and kept in a cool room at temperature of 4°C. Sample waste was characterized for organic and metal analysis. The properties of sludge are presented in Table I. The sludge has high volatile solid of 92 % which typically consisted of aromatic and semivolatile compounds. These compounds are detected in form of TPH of organic fraction.

B. Binder Materials

The binder used in the study was ordinary Portland cement based on Malaysian Standard 522 Part 1 2003 supplied by Lafarge Malaysia. The cement chemical composition is given in Table II. Cement replacement material used in the S/S was RHA which was prepared by slow burning in ferrocement furnace and grind to average size of less than 45 µm. It has a specific gravity of 2.01. Microstructure property of binder material was conducted using Philip EDAX XL30 scanning electron microscope (SEM) using gold palladium coating.

TABLE I. SLUDGE PROPERTY

Observation	Sludge Characterization		
	Parameter	*Value*	*Method*
Chemical/ physical [11]	pH	7.03	Photometric
	Specific gravity	0.96	APHA 2710F
	Moisture,%	53.80	APHA 2540G
	Oil & grease, mg/L	156.6	APHA 5520E
	Total solid, %	46.92	APHA 2540G
Organic fraction [12]	C6-C9, mg/kg	2030	5030B/8260B
	C10-C14, mg/kg	270000	3570/8015B
	C15-C28, mg/kg	537000	3570/8015B
	C29-C36, mg/kg	21900	3570/8015B

TABLE II. CHEMICAL COMPOSTION OF BINDER

Compound	Compound Concentration	
	OPC (%)	*RHA (%) [13]*
CaO	64.96	0.8
SiO_2	22.41	92.7
Al_2O_3	4.55	1.7
MgO	3.25	0.1
Fe_2O_3	3.15	0.4
SO_3	2.28	-

IV. TEST METHODS

S/S process involved three subsequent steps of mixture proportioning, casting and curing, and testing of solidified sludge performance. Mixture proportioning was carried out to find the best proportions based on the composition of cement to sludge ratio (C/Sd) at water to cement ratio (W/C) of 0.4, 0.45 and 0.45. The strength test has the objective to find long term durability of solidified waste subjected to appropriate pressure in the landfill site. Permeable porosity was tested to find the relationship with strength and mix design ratio.

A. Strength Test

Mould used for casting unconfined compressive strength (UCS) block was 50x50x50 mm according to ASTM C109-91[14]. Solidified sludge was cured in curing cabinet with moistened air at relative humidity of > 64 %. The UCS test was performed using a universal compression testing machine which complied with BS 1881: Part 116.

B. Permeable Porosity

Porosity was measured based on ASTM C642 [15] for measuring void in hardened concrete. A cylindrical sample of 350 cm^3 with diameter of 10 cm was used for the test. A dry sample was used to determine its mass. The percentage of permeable porosity or voids in the monolith was calculated based on Equation 4. The measurement of permeable porosity of solidified waste with RHA was extended to vacuum porosity and wet porosity as comparison [16].

Permeable porosity (voids), % = (g1-g2) / g2 x 100(4)

Where
g1 is dry bulk density in Mg/m^3
g2 is apparent density in Mg/m^3.

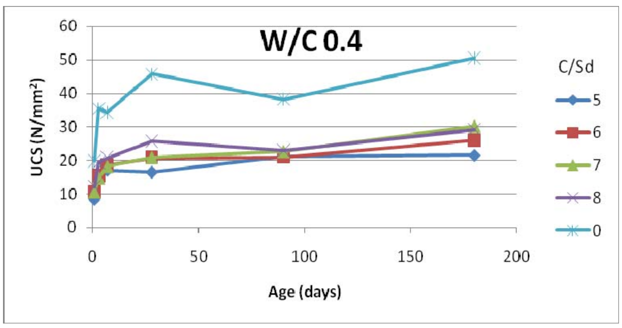
V. Results And Discussion

A. Compressive Strength

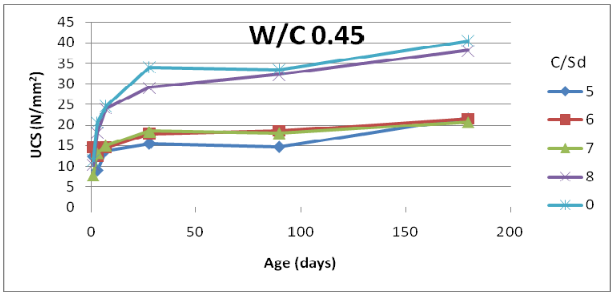
The UCS of solidified sludge was measured based on water to cement ratio (W/C) of 0.4, 0.45 and 0.5. The strength development of the solidified material is presented in Figure 1. The strength development of 0.45 W/C at C/Sd of 8 follow closely that of control OPC as illustrated in Figure 1(b) compared to other curves. The 180-day UCS was 38.2 N/mm^2 while 40.4 N/mm^2 was recorded for the solidified OPC. Thus the design parameters of W/C=0.45 and C/Sd=8 were used as base parameter for the cement replacement material study. Increasing water content in the mixture will reduce the strength. But lack of water affects workability and mouldability of samples.

Organic materials can be difficult to stabilize with inorganic binder because they interfere with setting reaction [17]. Organic interference with the cement hydration is due to the adsorption of contaminant on cement surface that block normal hydration process. Other interference mechanisms include complexation of aluminate, ferrite and silicate, delaying the formation of hydration products [18]. Nucleation inhibition can occur by organic as well as soluble silica or precipitating products of phosphates, borates and oxalates. Substituted aromatic undergo free radical oxidation [19].

Higher SO_3 content in OPC can cause the formation of ettringite at later ages and this can result in cracking in solidified waste which degrades mechanical properties and leaching behavior of waste forms [20]. Sodium hydroxide increased the rate of setting but reduce the strength of Portland cement. The presence of sodium hydroxide in solidified OPC-sludge is reflected by rapid increase in strength at early hydration period and followed by a sharp bending of UCS exactly at 28-day before slightly reduce to form almost plateau strength line. Sodium hydroxide as pH adjuster may solubilize silica for quicker reaction [21].



(a)



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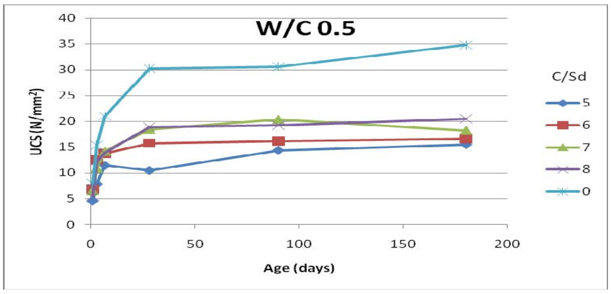


(c)

Figure 1. UCS evolution for three batches of OPC-sludge at W/C of (a) 0.4, (b) 0.45 and (c) 0.5.

B. Compressive Strength Analysis

The correlation anaylsis was executed by MINITAB 14 (MINITAB Inc., Pennsylvania, USA) for UCS values with age and C/Sd. The UCS has non linear relationship with age. Correlation is determind by Pearson correlation. UCS has significant correlation with age. Fitted line was plotted using quadratic regression based on Power model [22] for two variables of LogUCS as response and logAge as predictor. The relationship of both variables are represented by Equation 5, 6 and 7 for the three batches of W/C. The correlation and its P value, standard deviation (S), multiple determination coefficient (R^2) and adjusted R^2 for the three batches of OPC mixes are tabulated in Table III.

$$\text{W/C0.4: LogUCS} = 1.03 + 0.13\ \text{LogAge} - 0.01\ \text{LogAge}^2 \quad (5)$$

$$\text{W/C0.45: LogUCS} = 1.03 + 0.21\ \text{LogAge} - 0.03\ \text{LogAge}^2 \quad (6)$$

$$\text{W/C0.5: LogUCS} = 0.78 + 0.21\ \text{LogAge} - 0.02\ \text{LogAge}^2 \quad (7)$$

TABLE III. Statistical Analysis of OPC-Sludge

Observation	Statistical Parameter		
	W/C	***Value***	***Method***
Correlation of LogUCS, LogAge and SqrtC/Sd	0.4	UCS:Age 0.87(0.00) UCS:C/Sd 0.30 (0.14)	Pearson Correlation (P value)
	0.45	UCS:Age 0.73 (0.00) UCS:C/Sd 0.42(0.04)	
	0.5	UCS:Age 0.83 (0.00) UCS:C/Sd 0.28(0.17)	
LogUCS and LogAge	0.4 0.45 0.5	S, R^2(%), Adj. R^2(%) 0.06, 82.1, 80.4 0.11, 53.7, 49.3 0.07, 83.3, 81.7	Fitted line Power
Regression of LogUCS	0.4	S, R^2(%), Adj. R^2(%) 0.05, 86.0, 84.7	Multiple Regression

Observation	Statistical Parameter		
	W/C	*Value*	*Method*
with LogAge and SqrtC/Sd	0.45 0.5	0.09, 70.4, 67.6 0.08, 78.6, 76.6	

The multiple regression equations for the three parameters namely LogUCS, LogAge and SqrtC/Sd for the three batches of W/C are given by Equations 8, 9 and 10. Empirical model was developed for all parameters with linear relationship of UCS as response to age and C/Sd [23].

$$\text{W/C 0.4: LogUCS} = 0.58 + 0.06\ \text{LogAge} + 0.19\ \text{SqrtC/Sd} \tag{8}$$

$$\text{W/C 0.45: LogUCS} = 0.27 + 0.14\ \text{LogAge} + 0.30\text{SqrtC/Sd} \tag{9}$$

$$\text{W/C 0.5: LogUCS} = 0.73 + 0.08\ \text{LogAge} + 0.003\ \text{SqrtC/Sd} \tag{10}$$

C. RHA Cement Replacement Material

RHA was incorporated in solidified sludge mixture since the material is easily available in a rice growing nation such as Malaysia. The RHA was used at 5, 10 and 15 % as cement replacement material (CRM) of OPC. Incorporation of RHA showed a positive effect to the strength values compared to the control value (Figure 2). The usage of RHA was effective at 5 % replacement, based on the UCS of 25.0 N/mm^2 at 90-day compared to control sample of 19.2 N/mm^2. The U.S. EPA considers a stabilized material as satisfactory if it has UCS of 0.34 N/mm^2 or better.

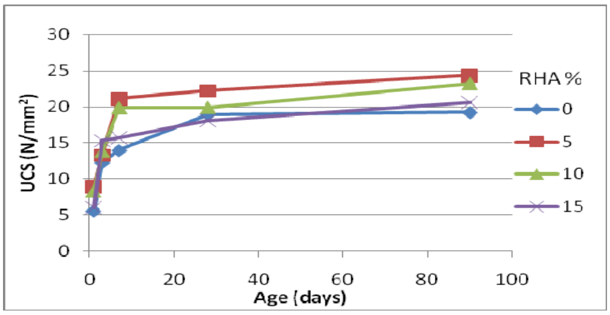


Figure 2. UCS development of 5, 10 and 15 % RHA OPC-sludge

D. Permeable Porosity

The porosity increased with the increases of cement content in the mixture as shown in Figure 3. The organic contaminants occupied the cement pore voids and this resulted in lower percentage of permeable pore in the solidifed sludge. Increasing RHA content also increased the void porosity in solidifed sludge due to its high silica content that create nucleation similar to that of organic material which inhibit cement hydration as seen in Figure 4. Vacuum porosity curve is almost similar with permeable porosity. Smallest void in C-S-H was 0.50 to 2.50 nm which account for 27 % of porosity, but gives little effect on stength. Capillary void in well hydrated low W/C was 10 to 50 nm where as 3000 to 5000 nm was measured at high W/C ratio. Capillary void of < 50 nm is detrimental to strength. Air void was in the range 0.05 to 0.20 mm, normally introduced to increase freeze-thaw resistance damage of monolith.

Hydrated cement exposed to CO_2 will produce calcite and silica gel, which can incorporate metals. Changes in chemical and physical preperties such as decrease porosity and shrinkage cause cracking to monolith are closely associated with atmospheric carbonation [24]. RHA sludge was found cracked after the porosity test. Since the sludge contained carbon compound, solidified RHA cement absorbed oxygen during the test and formed calcite and silica gel. Abundant silica provided by the RHA increases the carbonation products.

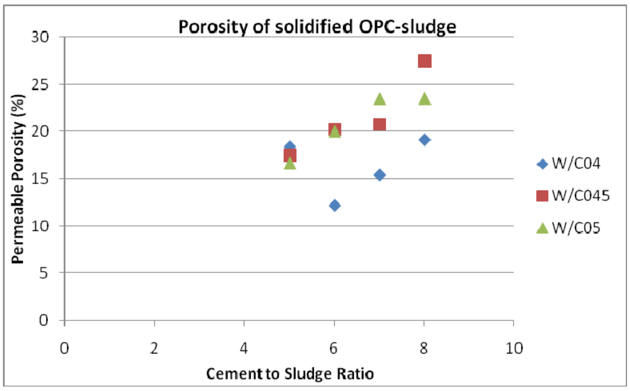


Figure 3. Porosity of solidified OPC-sludge

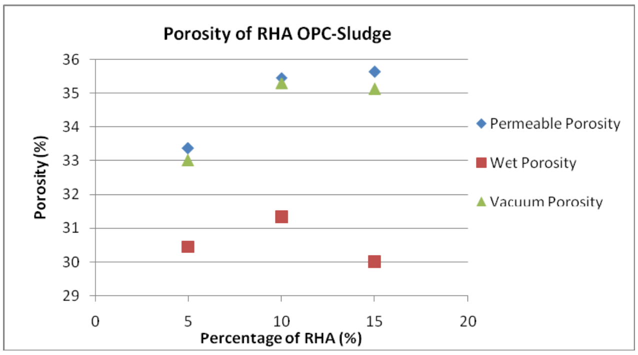


Figure 4. Porosity of RHA OPC-sludge

E. Relationship of Strength and Porosity

The relationship of strength with W/Sd and permeable porosity was plotted by Surfer 8.05, 2004 (Golden Software Inc. Colorado USA). The relationship between parameters were plotted on wireframe map overlay with contour map showing isostrength curve. The strength has inverse relationship with porosity for W/C of 0.4 as illustrated in Figure 5. The inverse relationship between strength and porosity is described by Equation 11 [7]. The planar regression equation for W/C=0.4 has coefficient of multiple determination (R^2) of 0.91.

$$S = S_0 e^{-kp} \tag{11}$$

Where S = strength of material with a given porosity p
S_0 = intrinsic strength at zero porosity
k = constant.

The strength and porosity has different trend for

W/C=0.45 where the increase in porosity resulted in increase of strength as illustrated in Figure 6. The trend is not in agreement with Equation 11, which is applicable for low W/C. R^2 of the regression equation was 0.95. The positive relationship between strength and porosity is due to the stronger cement structure formed.

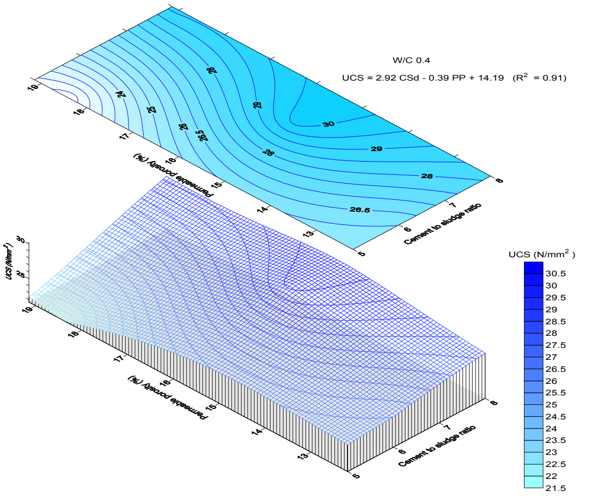


Figure 5. Relationship of strength with cement to sludge ratio and porosity at water to cement ratio of 0.4

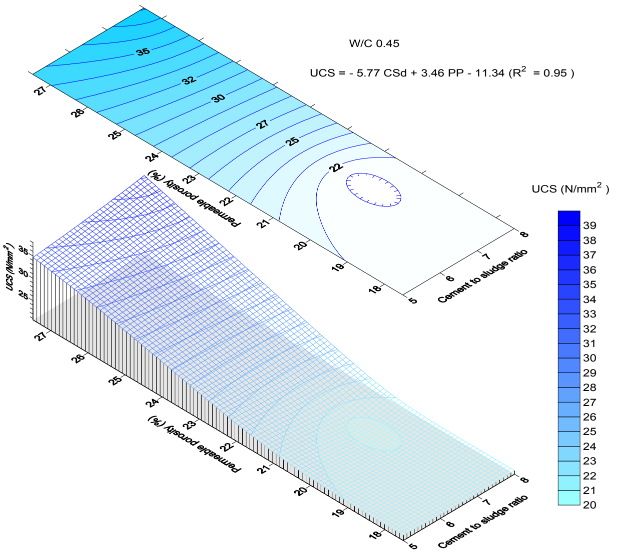


Figure 6. Relationship of strength with cement to sludge ratio and porosity at water to cement ratio of 0.45

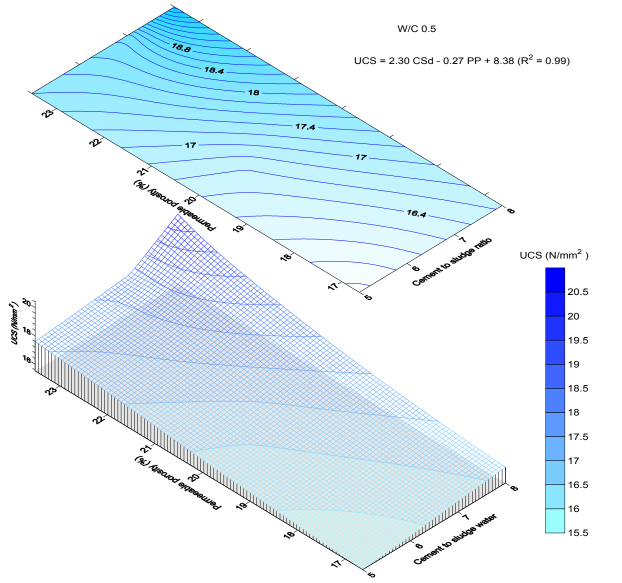


Figure 7. Relationship of strength with cement to sludge ratio and porosity at water to cement ratio of 0.5

Strength and porosity of W/C=0.5 were found to maximize at C/Sd of 8 with regression equation R^2 of 0.99 as illustrated in Figure 7. Based on the three batches of W/C, solidified waste with W/C=0.45 showed a good relationship of porosity and strength. Incorporation of more RHA in solidified waste as in Figure 8, cause reduction in strength but slightly increase in porosity was observed.

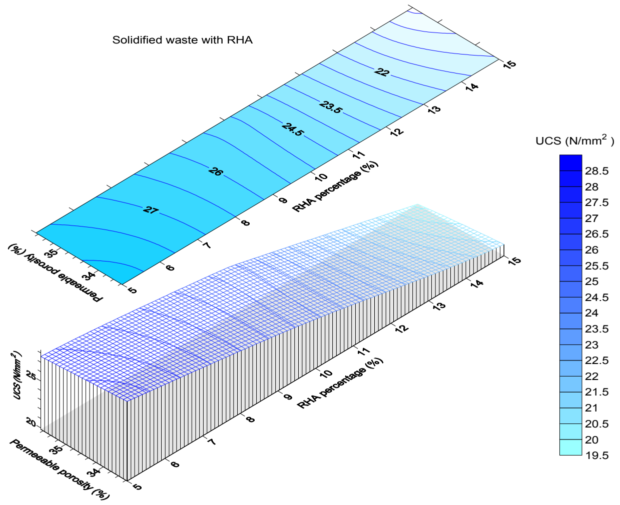
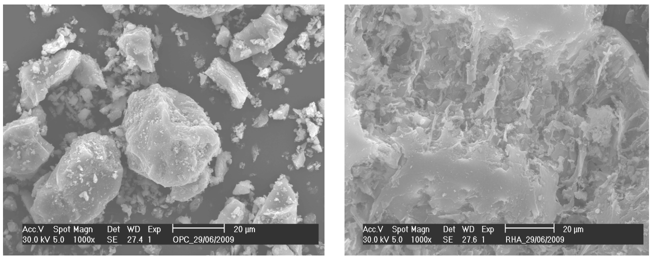


Figure 8. Relationship of strength with RHA percentage and permeable porosity

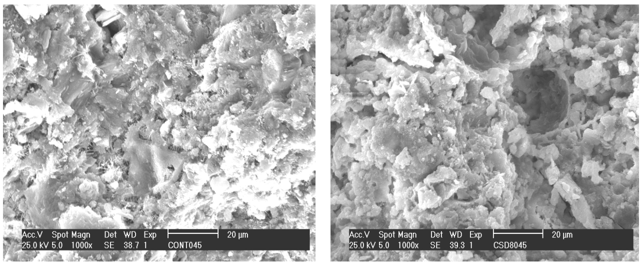
F. Microstructure of Solidified Sludge

Raw specimen of materials (OPC clinker and RHA) used in the S/S are depicted in Figure 9. The solidified sludge morphology captured at the age of 28 days showed that the oily sludge was absorbed by the amorphous hydrated cement products of C-S-H as illustrated in Figures 10. Addition of RHA has modified the surface morphology of the solidified sludge. More voids in the size range of 10 to 15 µm were clearly observed in Figure 11. Fewer voids were seen in RHA5 sample compared to RHA15 which can be contributed to the increased strength of the monolith.



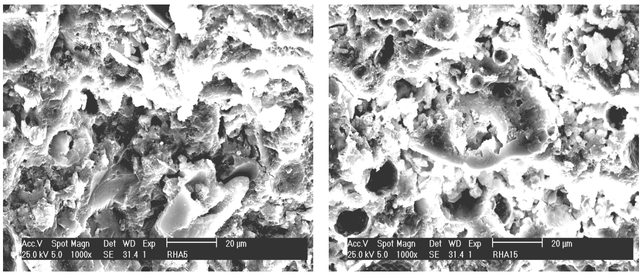
(a) (b)

Figure 9. SEM Micrographs x1000 of raw (a) OPC and (b) RHA



(a) (b)

Figure 10. SEM Micrographs x1000 of solidified (a) OPC (b) OPC-sludge



(a) (b)

Figure 11. SEM Micrographs x1000 of solidified OPC-sludge (a) RHA5 and (b) RHA15

VI. Conclusion

The strength development in the OPC-sludge is controlled by the W/C ratio. Sample of W/C ratio of 0.45 gave the highest strength at C/Sd of 8. These two values were selected as design parameters to incorporate RHA as CRM. In general, the addition of RHA was found to increase the strength of the solidified waste. The most effective RHA content was found at 5 %. At higher percentage, RHA modified the solidified sludge to a porous structure by inhibiting cement hydration and caused reduction in strength of monolith. This effect is supported by the trend in permeable porosity and vacuum porosity of RHA OPC-sludge which was subjected to moderately high temperature and pressure. This is due to the higher silica content in RHA which form soluble silicate and retard the tricalcium silicate hydration by nucleation mechanisms. Organic contaminants have also retarded the S/S system by adsorbing onto the surface of cement which hinders normal hydration reaction. The W/C and porosity are important factors that determine the strength of solidified waste. Increase in W/C ratio of the solidified sample resulted in the increasing porosity.

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