การใช้แกลบผงเพื่อเป็นสารเติมแต่งสำหรับซีเมนต์ในการเจาะหลุมปิโตรเลียม

นายณัฐพล แข้โส

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต

สาขาวิชาเทคโนโลยีธรณี

มหาวิทยาลัยเทคโนโลยีสุรนารี

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RICE HUSK ASH AS ADDITIVE FOR CEMENT

IN PETROLEUM WELL DRILLING

Nattapon Kaeso

A Thesis Submitted in Partial Fulfillment of the Requirements for the

Degree of Master of Engineering in Geotechnology

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RICE HUSK ASH AS ADDITIVE FOR CEMENT

IN PETROLEUM WELL DRILLING

Suranaree University of Technology has approved this thesis submitted in partial fulfillment of the requirements for a Master's Degree.

Thesis Examining Committee

____

(Prof. Dr. Kittitep Fuenkajorn)

Chairperson

____

(Asst. Prof. Dr. Akkhapun Wannakomol)

Member (Thesis Advisor)

____

(Assoc. Prof. Kriangkrai Trisarn)

Member

____

(Prof. Dr. Sukit Limpijumnong)

Vice Rector for Academic Affairs

and Innovation

____

(Assoc. Prof. Flt. Lt. Dr. Kontorn Chamniprasart)

Dean of Institute of Engineering

ณัฐพล แข้โส : การใช้แกลบผงเพื่อเป็นสารเติมแต่งสำหรับซีเมนต์ในการเจาะหลุมปิโตรเลียม (RICE HUSK ASH AS ADDITIVE FOR CEMENT IN PETROLEUM WELL DRILLING) อาจารย์ที่ปรึกษา : ผู้ช่วยศาสตราจารย์ ดร.อัมพรรค์ วรรณโกมล, 105 หน้า

วัตถุประสงค์หลักของการศึกษานี้ คือ การศึกษาการใช้แกลบผงเป็นสารเติมแต่งของซีเมนต์ที่ใช้กับหลุมน้ำมัน และศึกษาคุณสมบัติทางกายภาพของซีเมนต์ซึ่งถูกผสมด้วยแกลบผงประกอบด้วยความแข็งแรงในการต้านทานแรงกดของซีเมนต์ที่แข็งตัวแล้ว และคุณสมบัติทางวิทยากระแสของซีเมนต์เหลวที่ผสมแกลบผง ในการศึกษานี้ได้ใช้ซีเมนต์ที่ใช้กับหลุมน้ำมันคลาสจีและแกลบผงที่ได้ถูกรวบรวมมาจากในพื้นที่ และถูกจัดเตรียมสำหรับการทดสอบตัวอย่างซีเมนต์เหลวโดยถูกทดแทนด้วยแกลบผงที่ร้อยละ 5 10 15 และ 20 โดยน้ำหนัก และนำมาวัดค่าความหนาแน่นความหนืด และปริมาณของไหลที่สูญเสีย ตัวอย่างของซีเมนต์เหลวบางส่วนถูกใช้สำหรับการทำตัวอย่างซีเมนต์แข็ง ตัวอย่างซีเมนต์แข็งได้ถูกบ่มที่อุณหภูมิ 25 และ 80 องศาเซลเซียส ด้วยเวลาบ่ม 3 7 14 28 และ 56 วัน ตามลำดับ ตัวอย่างซีเมนต์แข็งที่เวลาบ่มต่างๆ ได้ถูกนำมาทดสอบความแข็งแรงในการต้านทานแรงกด และวัดค่าความซึมซาบ ผลการทดสอบและตรวจวัดชี้ให้เห็นว่าค่าความแข็งแรงในการต้านทานแรงกดของตัวอย่างซีเมนต์แข็งนั้นเพิ่มขึ้นเมื่อปริมาณของแกลบผงและเวลาที่ใช้บ่มนั้นเพิ่มขึ้นอันเนื่องมาจากอิทธิพลของปฏิกิริยาพอซโซลานิก ค่าความหนืดและปริมาณของไหลที่สูญเสียของซีเมนต์เหลวที่ผสมแกลบผงนั้นเพิ่มขึ้นเมื่อปริมาณของแกลบผงเพิ่มขึ้นในขณะที่ความหนาแน่นของซีเมนต์เหลวผสมแกลบผงกลับลดลง อย่างไรก็ตามสามารถสรุปได้ว่าแกลบผงสามารถใช้เป็นสารเติมแต่งของซีเมนต์ที่ใช้กับหลุมน้ำมันได้ ถ้ามันถูกใช้แทนที่ซีเมนต์ที่ใช้กับหลุมน้ำมันที่ร้อยละ 10 โดยน้ำหนัก ทั้งนี้เพราะว่าการลดลงของความหนาแน่นและปริมาณการสูญเสียของไหลของซีเมนต์เหลวนั้นไม่แตกต่างไปจากซีเมนต์ที่ไม่ได้ผสมแกลบผงมากนักในขณะที่ค่าความแข็งแรงในการต้านทานแรงกดนั้นสูงกว่า

สาขาวิชา <u>เทคโนโลยีธรณี</u>	ลายมือชื่อนักศึกษา_______________________
ปีการศึกษา 2558	ลายมือชื่ออาจารย์ที่ปรึกษา_______________

NATTAPON KAESO : RICE HUSK ASH AS ADDITIVE FOR CEMENT IN PETROLEUM WELL DRILLING. THESIS ADVISOR : ASST. PROF. AKKHAPUN WANNAKOMOL, Ph.D., 105 PP.

RICE HUSK ASH/ CEMENT ADDITIVE/ COMPRESSIVE STRENGTH/ RHELOGICAL PROPERTIES ENCHANCEMENT

The main objectives of this study are to study the using of rice husk ash (RHA) as an additive of the oil well cement and study the physical properties of cement mixed with RHA, including the compressive strength of set cement and rheological properties of the RHA cement slurry. In this study oil well cement class G was used and RHA was collected from local area and prepared for testing. Cement slurry samples were replaced by RHA at 5, 10, 15 and 20 by weight percent, and were measured to determine density, viscosity, and filtrate loss volume. Some cement slurry samples were used for making set cement specimens. Set cement specimens were cured at 25°C and 80°C with curing times of 3, 7, 14, 28, and 56 days, respectively. Set cement specimens at various curing time were tested to determine compressive strength and were measured their permeability. Result of the tests and measurements indicated that the compressive strength of set cement specimens was increased with the amount of RHA and curing time increasing due to the effect of pozzolanic reaction. Viscosity and filtrate loss volume of the RHA cement slurry was increased with the amount of RHA increasing, while density of the RHA cement slurry was decreased. However, it can be concluded that RHA can be used as an oil well cement additive if it is used to replaced oil well cement at 10 percent by weight.

This is because the reduction of density and filtrate loss volume of the cement slurry are not much different from the cement without RHA while the compressive strength is higher.

School of Geotechnology Student's Signature__________________

Academic Year 2015 Advisor's Signature__________________

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Nattapon Kaeso

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SYMBOLS AND ABBREVIATIONS

γ	=	shear rate, (sec^{-1})
τ	=	shear stress, (lb_f/ft^2)
ϕ_{300}	=	viscosity dial reading at 300 rpm
ϕ_{600}	=	viscosity dial reading at 600 rpm
μ	=	gas viscosity, (cp)
μ_a	=	apparent viscosity, (cp)
μ_p	=	plastic viscosity, (cp)
ϕ_i	=	viscometer dial reading
γ_p	=	Yield point, ($lb_f/100ft^2$)
A	=	cross-section area, (cm^2)
K	=	fluid consistency index, (md)
K	=	Permeability, (md)
L	=	sample length, (cm)
N	=	flow behavior index
N	=	range extension factor of the torque spring of the VG meter
P_1	=	inlet pressure, (atm)
q	=	flow rate, (cm^3/sec)
t	=	time of blow out, (min)
V_t	=	volume of filtrate collected at the time of the blowout, (ml)
BP	=	barometric pressure, (atm)

CHAPTER I

INTRODUCTION

1.1 Background and rational

In petroleum drilling industry cement is filled and sealed between the casing string and the drilled hole for three general purposes:

(1) Zone isolation and segregation

(2) Corrosion control

(3) Formation stability and pipe strength improvement

In petroleum well drilling industry cement is a material used in a large amount and quite expensive. One approach to reduce cost in petroleum well drilling methodology is mixing cement with additive, such as silica fume, lignosulfonates, gilsonite etc. However these additives are expensive and have to be imported from aboard. Rice Husk Ash (RHA) is one of the alternative additives that can be used as a cement extender additive. Rice husk ash is a by-product from the combustion of rice husk which is available in large number in Thailand. Because of RHA is light weight and has CaO and SiO_2 as major composition, it is used as a cement extender additive in building construction as presented by many authors e.g. Dabai et al. (2009), Hwang et al. (2011), Karim (2012), and Jawad and Alwash (2013). The objective of this study is aimed to use of RHA as a cement extender additive for the cement which is used in petroleum well drilling since it is abundant, low cost, and widely available in Thailand. Though there are many researchers studied on using RHA as a cement additive, most of them was focused on cement strengthening after mixing with RHA. Therefore, this research did not study only about the effect of RHA on oil well drilling cement strength and thickening time, but also study about the rheological properties of RHA cement. They are very important to the cement replacement in petroleum well drilling, e.g. thickening time and cement replacement time.

1.2 Research objectives

The main objectives are to study the using of rice husk ash (RHA) as an additive of the oil well cement and to study the physical property of cement mixed with RHA, including the compressive strength of set cement and rheological properties of RHA cement slurry.

1.3 Research methodology

1.3.1 Literature review

The relevant literatures from books, journals, and previous works are searched and reviewed. The summary of the literature review was given in this thesis.

1.3.2 Cement samples preparation

API class G cement were prepared as cement slurry mixed with RHA at various weight percent ratio at 5, 10, 15, and 20 respectively and these mixed cement then had been set according to API standard. Some cement slurry specimens had been prepared for rheology test, including cement density, permeability and gel strength test respectively. Some set (solid) cement specimens had been prepared for cement compressive strength test and thickening time test, respectively.

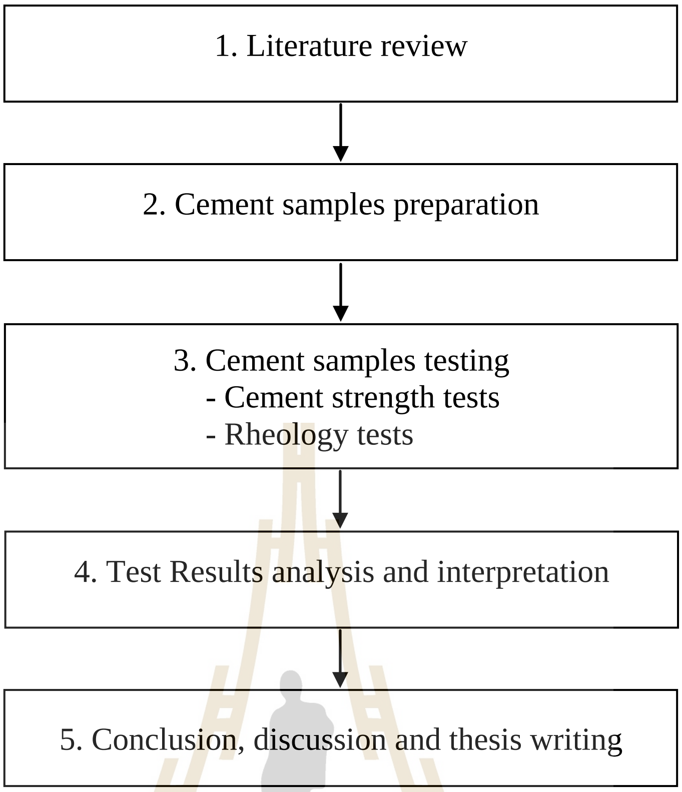


Figure 1.1 Research methodology

1.3.3 Cement samples testing

1.3.3.1 Set cement strength test and thickening time test

1.3.3.1.1 Compressive Strength: Set cement specimens had been cured at 25°C (77°F) and 80°C (176°F) for the designed testing period. Specimens then had been tested by compressive strength testing machine.

1.3.3.1.2 Thickening time: A thickening time test had been conducted by Vicat apparatus. That was measured about setting time of cement.

1.3.3.2 Rheology

1.3.3.2.1 Density: Slurry density had been tested by Pressurized fluid density balance.

1.3.3.2.2 Permeability: Permeability measurement of cement specimences had been conducted according to the Darcy's Law by using permeameter.

1.3.3.2.3 Gel Strength: The gel strength of cement slurry were be measured by using viscometer.

1.3.3.2.4 Fluid-loss test: Fluid loss test was performed according to API RP-10B and some of them were based on standard ISO filter press test in which filtration pressure was 100 psi and support screen 60 mesh were used as the filtration area.

1.3.4 Test results analysis and interpretation

The research results were analyzed and interpreted to compare the compressive strength, permeability, fluid loss, gel strength, and thickening time between cement which were mixed with RHA in various ratios.

1.3.5 Conclusion, discussion and thesis writing

Discussions of the results were described to determine the reliability and accuracy of the measurements. Performance of the RHA cement extender additive was discussed based on the test results. All research activities, methods, and results were documented and complied in the thesis.

1.4 Scope and limitations of the study

The study was scoped and tested only the API class G cement (API) due to it is an ordinary cement type and it is mainly used in shallow well drilling which temperature is not a critical factor.

1.5 Thesis contents

Chapter I introduces the thesis by briefly describing the background of problems and rational. The research objectives, methodology, scope and limitations of the study are also identified in chapter I. **Chapter II** summarizes the results of the literature review. **Chapter III** describes the sample preparations and test methods. **Chapter IV** describes and discusses the results from the laboratory experiments. **Chapter V** discusses and concludes the research results and gives some recommendations for the future experiments.

CHAPTER II

LITERATURE REVIEW

2.1 Introduction

This chapter summarizes the results of literature review which can be divided into two main topics:

(1) Cement in petroleum drilling industry, including cement chemical properties, cement physical properties and cement additives.

(2) Rice Husk Ash (RHA)

2.2 Cement in petroleum drilling industry

Most cement used in the petroleum industry is a type of Portland cement. Thomas and Alan (2006) explained this type of cement is used in well drilling for 5 purposes as;

(1) To isolate the hydrocarbon bearing formations from water bearing formations and one hydrocarbon bearing formation from another, i.e, gas and oil,

(2) To protect and secure the casing in the hole,

(3) To prevent caving of the hole,

(4) To provide a firm seal and anchor for the wellhead equipment, and

(5) To protect casing from corrosion by sulfate-rich formation waters, if exposed to direct contact with the casing.

Portland cement is produced from limestone and other materials (such as clay or shale) by heating at 2,600°F to 3,000°F (Thomas and Alan, 2006).

2.2.1 Cement chemical properties

Portland cement contains of two Oxide groups as;

(1) Major Oxide: CaO, SiO_2, Al_2O_3, and Fe_2O_3 about 90% of the total weight of the cement.

(2) Minor Oxide: MgO, Na_2O, K_2O, TiO_2, P_2O_5 and Gypsum.

Oxides which are components of Portland cement are shown in Table 2.1.

Table 2.1 Major and minor oxide components of Portland cement (modified after บริษัทปูนซีเมนต์ไทยจำกัด, 2549)

Oxide		**Percent by weight**
Major Oxide	CaO	60 - 67
	SiO_2	17 - 25
	Al_2O_3	3 - 8
	Fe_2O_3	0.5 - 6.0
Minor Oxide	MgO	0.1 - 5.5
	$Na_2O + K_2O$	0.5 - 1.3
	TiO_2	0.1 - 0.4
	P_2O_5	0.1 - 0.2
	SO_3	1 - 3

Major and minor oxides are assembled during clinking and 8 main compounds are then produced as shown in Table 2.2.

Table 2.2 Main compounds of major and minor oxide of Portland cement (modified after บริษัทปูนซีเมนต์ไทย จำกัด, 2549)

Main Compound		Chemical Composition	Abbreviation
Major Oxide	Tricalcium Silicate	$3CaO \times SiO_2$	C_3S
	Dicalaium Silicate	$2CaO \times SiO_2$	C_2S
	Tricalcium Aluminate	$3CaO \times Al_2O_3$	C_3A
	Tetracalcium Aluminoferrite	$4CaO \times Al_2O_3 \times Fe_2O_3$	C_4AF
Minor Oxide	Calcium Salphate Dihydrate (Gypsum)	$CaSO_4 \times 2H_2O$	-
	Free Lime	-	CaO
	Magnesium Oxide	-	MgO
	Alkali Oxides	-	Na_2O, K_2O

API basically divided cement into class A to class J (without class I). These classifications of cement depends on deeper and hotter down hole conditions.

API Cement Classes (King, 2014)

Class A: For use from surface to 6,000 feet (1830 meter) depth when special properties are not required.

Class B: For use from surface to 6,000 feet (1,830 meter) depth when conditions require moderate to high sulfate resistance.

Class C: For use from surface to 6,000 feet (1,830 meter) depth when conditions require high early strength.

Class D: For use from 6,000 to 10,000 feet (1,830 to 3,050 meter) depth when under conditions of high temperatures and pressure.

Class E: For use from 10,000 to 14,000 feet (3,050 to 4,270 meter) depth when under conditions of high temperature and pressures.

Class F: For use from 10,000 to 16,000 feet (3,050 to 4,880 meter) depth when under conditions of extremely high temperatures and pressures.

Class G: Intended for use as basic cement from surface to 8,000 feet (2,440 m) depth. Can be used with accelerators and retarders to cover a wider range of well depths and temperatures. Cement that use in this thesis is in this class.

Class H: A basic cement for use from surface to 8,000 feet (2,440 meter) depth as manufactured. Can be used with accelerators and retarders to cover a wider range of well depths and temperatures.

Class J: Intended for use as manufactured from 12,000 to 16,000 feet (3,600 to 4,880 meter) depth when under conditions of extremely high temperatures and pressures. It can be used with accelerators and retarders to cover a range of well depths and temperatures.

2.2.2 Cement physical properties

Portland cements are commonly characterized by their physical properties for quality control purposes. Their physical properties can be used to classify and compare Portland cements. The challenge in physical property characterization is to develop physical tests that can satisfactorily characterize key parameters, including compressive strength and pump ability time.

2.2.2.1 Compressive Strength

Compressive strength of neat cement increases over the period of several weeks. The compressive strength of particular time during the curing process depends heavily on mixing and curing conditions. Typical compressive strength of API Class G cement during various curing conditions is shown in Table 2.3.

2.2.2.2 Pump ability time

Pump ability time or thickening time of cement slurry is an important factor in slurry design. Generally these statements can be made regarding thickening time. Some conclusions on pump ability time and temperature, pressure, and water content are listed as follows.

- Higher the temperature – faster the set. This is a primary factor affecting thickening time.

- Higher the pressure – faster the set. This effect is more pronounced below 5000 psi, but continues up through limits of lab test equipment.

- Loss of water from slurry accelerates set.

- Shutdown during placement results in cement gellation which will shorten pump ability and accelerate set and strength development.

Table 2.3 Typical compressive strength of API class G cement during various curing conditions (after Thomas and Alan, 2006)

Curing conditions		24-hour compressive strength, psi
Temp (°F)	**Pressure (psi)**	
80	0	615
60	0	1,470
95	800	2,085
110	1,600	2,925
140	3,000	5,050
170	3,000	5,920

The effect of temperature on thickening time of API class G cement is presented in Table 2.4.

Table 2.4 Relations between thickening time and temperature of API class G cement (modified after Thomas and Alan, 2006.)

Circulating Temperature (°F)	Thickening Time (hours : minute)
91	4:00+
103	3:36
113	2:25
125	1:40

2.2.3 Cement and additives

The raw ingredients of Portland cement are lime, silica, alumina and iron oxide. Today's well cements have to resist a big range of well depths and conditions. In permafrost zones, the cement must resist below-freezing condition, while in thermal recovery wells or geothermal fields they must endure temperatures above 350°C (660°F).

In petroleum drilling industry have varied conditions, cement can be formulated by using additives. Cement additives which number more than 100 and are used for different profits. However cement additives can be grouped into 8 major categories as follows:

1. Accelerators: reduce cement setting time and speed up the development of compressive strength. They are commonly used in shallow, low temperature wells.

2. Retarders: extend cement setting time and allow sufficient time for slurry placement in deep wells.

3. Extenders: reduce cement density and may also reduce the amount of cement needed for a job. Low-density cement is needed for cementing weak formations which would otherwise breakdown and cause lost circulation.

4. Weighting agents: increase cements density. These are used for cementing high-pressure formation which might become unstable if slurry density were too low.

5. Dispersant: reduce the viscosity of cement slurry and ensure good mud removal during placement.

6. Fluid-loss control agents: control water loss from the cement into the formation.

7. Lost – circulation control agents: reduce the loss of cement slurry into weak or vuggy formations. Loss of cement may necessitate a costly, remedial cementing operation.

8. Special additives such as antifoam agents and fibers, are manufactured for specific cementing tasks, such as the prevention of foaming that might lead to a loss in hydraulic pressure. (อัฆพรรค์ วรรณโกมล, 2555)

In this study the rice husk ash (RHA) was tested and used as a cement extender additive.

2.3 RHA

Rice husk is the hard protecting coverings of rice grain. Rice husk ash is produced after the husk had been burned. One of the major compositions of RHA is silica, which is a substance that can accelerate chemical reactions in the oil and petrochemical industry. Some literatures about physical properties, chemical properties, and using of RHA as an additive of construction cement are summarized as follows.

2.3.1 Physical properties

2.3.1.1 Density

Densities of RHA depend on method of combustion, if combustion is not completed, RHA density will be low since it is composed of high carbon content. Density of completed combustion range between 1.9 and 2.3 depended on the combustion temperature. If the combustion temperature is about

500°C, the RHA density is 2.06 g/cc. If the combustion temperature is raised up to 800°C and 1000°C, the density of RHA will be 2.20 and 2.30 g/cc because of the decreasing of the carbon contents (บุรฉัตร ฉัตรวีระ และทวิสัณห์ คงทรัพย์, 2545).

2.3.1.2 Pozzolanic of RHA

Pozzolanic of RHA can be measured from standard ASTM C311 (2013) by the compressive strength test at 7 and 28 days from mortar which is made from Portland cement mixed with of RHA 20 wt%. In general the compressive strength of the RHA pozzolanic cement is higher than those of pure Portland cement (Gemma, 2005).

2.3.2 RHA chemical composition

From the study of rice husk ash (RHA) chemical compositions (บุรฉัตร และทวิสัณห์, 2545) are as presented in Table 2.5.

2.3.3 Rice husk ash and cement

Rice husk ash (RHA) is mixed with cement in order to increase cement's volume and strength. There are some researches which studied about the using of RHA in cement mortar or concrete. Most of researches were studied mainly about compressive strength and setting time of the mixed cement as it can be summarized as followings.

Gemma (2005) studied on the development of compressive strength up to 91 days of concretes mixed with residual from rice puddy milling industry in Uruguay (UY) and the RHA produced by controlled incineration from USA were used for comparison. The result founded that concrete mixed with UY RHA presented higher compressive strength than those of concrete mixed with USA RHA

(Table 2.6). The RHA concrete also had higher compressive strength at 91 days than the concrete without RHA. It was also found that the compressive strength of cement mixed with USA RHA and UY RHA were significant different between 7 and 28 days curing time. It is noted that the increase in compressive strength of concretes with RHA produced by controlled incineration is mainly due to the pozzolanic effect.

Table 2.5 Chemical composition of rice husk ash (modified after บุรฉัตร และ ทวิสัณห์, 2545)

Composition	**Weight (%)**
Silicon dioxide (SiO_2)	78.120
Aluminum dioxide (Al_2O_2)	0.310
Iron dioxide (Fe_2O_3)	0.230
Calcium oxide (CaO)	0.080
Magnesium oxide (MgO)	0.034
Sodium oxide (Na_2O)	0.170
Potassium oxide (K_2O)	0.820
Titanium oxide (TiO_2)	0.040
Sulfur trioxide (SO_3)	0.09
Percent of loss weight by burning (LOI)	8.31

Table 2.6 Compressive strength test of cement mixed with RHA (after Gemma, 2005)

w/(c + RHA)	RHA Type	RHA %	f_c (MPa) 7d	f_c (MPa) 28d	f_c (MPa) 91d	$f_{t,d}$ (MPa) 28d	K_t (m^2) 28d
0.32		0	48.4	55.5	60.6	3.63	1.08×10^{-16}
	UY	10	51.1	60.4	64.3	3.57	0.23×10^{-16}
		20	44.3	54.8	62.7	3.34	0.05×10^{-16}
	USA	10	39.5	51.4	64.5	3.62	0.08×10^{-16}
		20	30.5	47.4	68.5	3.54	0.03×10^{-16}
0.40		0	35.8	42.3	45.6		
	UY	10	41.1	50.4	54.9		
		20	27.9	40.7	51.4		
	USA	10	29.7	40.8	51.5		
		20	23.6	39.4	57.3		
0.50		0	24.6	32.9	35.9	2.85	28.20×10^{-16}
	UY	10	24.1	31.5	35.5	2.32	71.82×10^{-16}
		20	24.9	34.9	37.9	2.63	49.10×10^{-16}
	USA	10	22.7	34.5	44.4	2.92	26.36×10^{-16}
		20	20.8	35.9	52.9	3.00	14.20×10^{-16}

Keys: f_c = axial compressive strength; $f_{t,d}$ = splitting tensile strength; K_t = permeability coefficient.

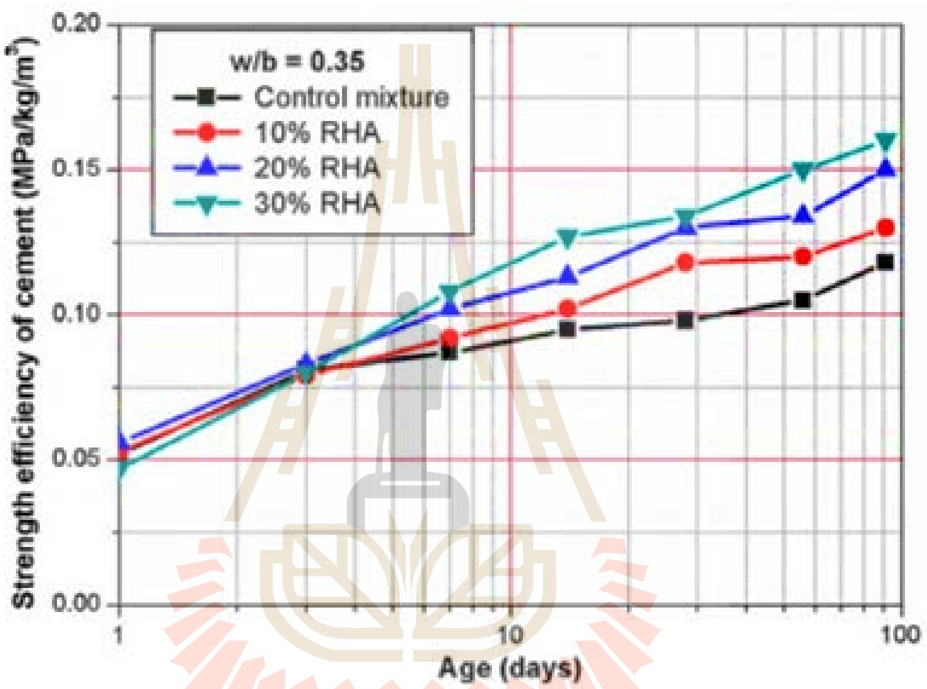
Dabai et al. (2009) studied on compressive strength, setting time, and chemical analysis of cement mixed with RHA. The chemical analysis of RHA result shows that RHA has high amount of silica (68.12%). For setting time testing, the increasing in setting time of paste having RHA shows low level of hydration for RHA concrete. This is resulted from the reaction between cement and water which liberate calcium hydroxide ($Ca(OH)_2$). Compressive strength test shown that the best compressive strength result was obtained from cement samples which were replaced by 10% RHA and it was decreased as the percentage of RHA was increased (Table 2.7). The compressive strength of specimens also increases with the setting time increasing as the highest compressive strength encountered at 28 days. This may be due to the retention of water with the structural framework of the mixture there by allowing.

Table 2.7 Compressive strength test of cement mixed with RHA (after Dabai et al., 2009)

Amount of Cement (%)	Amount of RHA(%)	Design Strength (N/mm^2)				
		1 Day	3 Days	7 Days	14 Days	28 Days
100	0	16.00	25.70	28.00	32..30	41.00
90	10	12.60	14.20	22..10	28.50	36.30
80	20	6.70	10.40	18.6 0	24.30	30.20
70	30	4.20	8.60	16.3 0	22.40	24.00
60	40	2.00	6.20	14.4 0	18.20	20.30
50	50	0.90	4.10	9.20	11.50	14.00

Hwang et al. (2011) compared the compressive strength between non-RHA and 1 hour ground RHA (from south Vietnam) cylindrical concrete. Results of the study indicate that factors influencing the strength of concrete are the types and quality of materials, the mixture proportion, the construction methods, the curing condition, and the test method. From the microscopic point of view, both the degree of hydration, and the porosity play important roles. The greater the volume of the pores, the lower the strength of the concrete will be. In addition, with decreasing of binder/space ratio (defined as the ratio of the content of C–S–H gel to the original volume of space), the strength will become greater. This is evident in Figure 2.1, which shows the relationship between testing age and compressive strength of concrete with various ground RHA content at the same and different w/b ratios. In the early phase, the addition of ground RHA reduces the amount of cement by 10–20%, the volume of capillary pores then increases, accumulating CH on the interface. As a result, the structure is less compact, causing the strength to be lower than that of the specimen without ground RHA added. After 28 days, pozzolanic reaction starts to proceed, which is resulted in decreasing of the amount of CH and improving of the densification. Consequently, the compressive strength is enhanced in the later phase.

Comparison of the data for 56 and 91 days of curing ages shows that the compressive strength of concretes with up to 20% ground RHA attain values equivalent to that control concrete specimens. With water-to-binder ratio from 0.23 to 0.47, compressive strength at 28 days of RHA concrete in the 47–66 MPa range is obtained in the investigation.



Mix	*w/b* ratio	RHA content (%)	Compressive strength (MPa)						
			1 day	3 days	7 days	14 days	28 days	56 days	91 days
A23-10	0.23	10	41	59	62	63	66	69	74
A35-00	0.35	0	30	46	50	54	56	60	67
A35-10	0.35	10	27	41	47	52	61	62	67
A35-20	0.35	20	26	38	47	52	60	61	69
A35-30	0.35	30	19	32	43	51	54	60	64
A47-10	0.47	10	16	26	37	40	47	51	56

Figure 2.1 Strength Development of compressive strength (Hwang et al., 2011)

Karim et al., (2012) studied on the strength of mortar and concrete as Influenced by RHA. In this experiment, RHA is used as supplementary cementing material in mortar and concrete and has demonstrated significant influence in improving the mechanical and durability properties of mortar and concrete. The result shown that the strength development of concrete produced with a particular level of RHA replacement is the same or higher as compared to Ordinary Portland Cement (OPC) concrete. At about 20-30 wt% RHA replacement, no significant reduction in strength of concrete can be observed. Moreover, a valuable cement and energy saving consideration could be performed by proper utilization of RHA in the production of cement or concrete industry.

Jinan and Alwash (2013) studied on using of Rice Husk Ash in cement mortar. This research focused on the effects of using RHA as partial cement replacement materials in cement mortar mixed. The study of mortar made with Ordinary Portland Cement (OPC) which were and replaced by different amount of RHA in dry condition. This work was conducted and tested with normal consistency, initial and final setting time, compressive strength, and flexural strength. Results indicated that RHA could significantly improve the cement mortar strength at the 15 wt% replacement level at the age of 90 days (Figure 2.2). The result also indicated that using RHA at the 5 wt%, 10 wt%, and 15 wt% replacement level decreased the porosity of cement mortar in comparison with controlled mortar samples at ages 7, 28, and 90 days. But at 20 wt% cement replacement level by RHA found that the porosity of mortars had been increased in all ages.

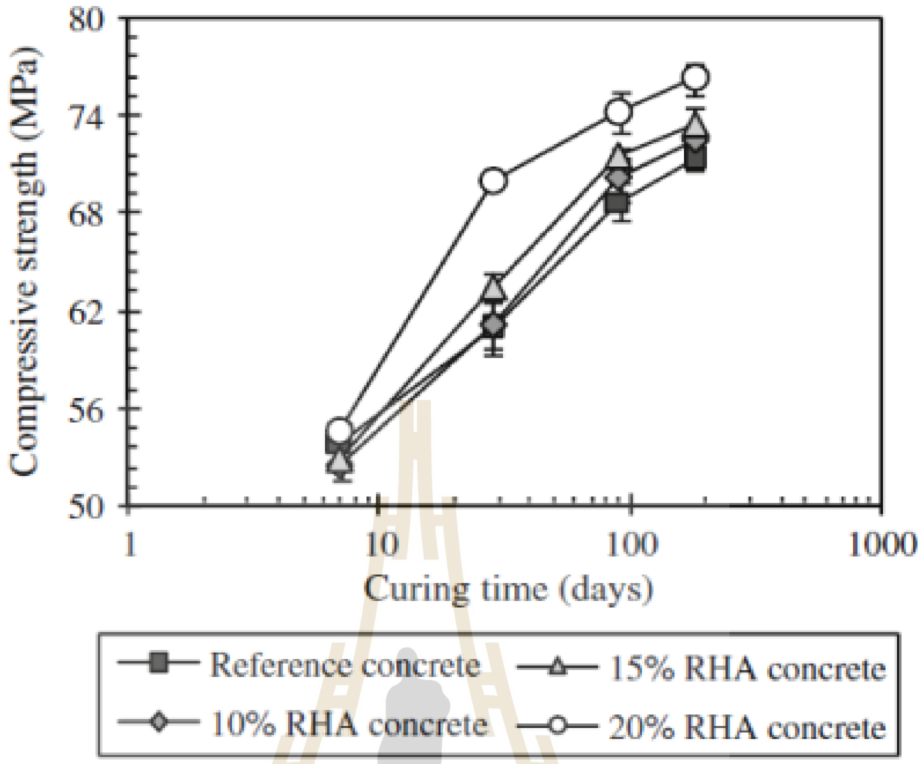


Figure 2.2 Compressive strength development of cement with various ratio RHA added (Jawad and Alwash, 2013)

Consequently, most of reviewed researches mainly focused only on compressive strength, and setting time testing of rice husk ash as an additive. Only a few studied on flexural strength, viscosity, and chemical analysis of cement replacement by RHA. Since RHA could be used as an additive for increasing the compressive strength of concrete as in previous mentioned, this study not only studied on the effect of RHA on the oil well cement strength and thickening time, but also studied on the physical, and rheological properties of RHA cement e.g. density, permeability, filtration, and shear strength. This is because these rheological properties are very important factor, and play important roles in cement replacement in an oil well drilling process.

CHAPTER III

RESEARCH METHODOLOGY

3.1 Introduction

This chapter describes basic characteristics of tested materials in this study which are RHA and well drilling cement, samples collection and preparation, cement specimens measurement and testing respectively.

3.2 Sample collection and preparation

3.2.1 Sample collection

3.2.1.1 RHA

RHA samples used in this research were been bought from Thai Naronk Kut Chik Limited Partnership. RHA samples were collected and packed in plastic bag. RHA was dried in a hot-air oven at 140°C for at least 24 hours at the Geotechnology Laboratory of Suranaree University of Technology, Nakhon Ratchasima province.

3.2.1.2 Cement

Oil well cement class G of the TPI POLENE Public Company Limited, Thailand, was used in this study since it was met according to the API (10A) standard. The cement was kept in plastic bag and sealed to prevent moisture and stocked in cool-dry area.

3.2.1.3 Cement sample mixing

Dry cement samples were mixed with water/binder ratio (W/B) at 0.5 and added RHA instead of cement at 0 wt%, 5 wt%, 10 wt%, 15 wt% and 20 wt%, respectively in the blending apparatus (Figure 3.1)

Figure 3.1 Blend machine set

3.3 Cement specimen measurement and test methods

3.3.1 Compressive strength

Set (solid) cement samples were prepared in cube mold 10 cm x 10 cm size. These samples were cured at 2 temperatures as 25°C and 80°C for 3, 7, 14, 28, and 56 days and dried before taking to the compressive strength test machine (Figure 3.2) at Civil Engineering Laboratory of Suranaree University of Technology. The test results were presented in maximum load of cement cube in Kilo-Newton unit.

Figure 3.2 Compress machines

3.3.2 Permeability

Sample Preparation

Set cement samples were prepared in cylinder mold having diameter 1.5 inch and length 2 inch and tested by Permeameter (Figure 3.3).

The sample core tube was full filled with the liquid and then gas was injected. Once a stabilised flow rate was established, recorded the Upstream pressure (P1, psig), Over burden pressure (Pc, psi), Flow volume (cm^3), flow time (second), Barometric pressure (atm), and temperature (celcius) from instrument. The injected gas permeability (Kg) can then be calculated by using Darcy's Law as presented in equation 3.1 and 3.2.

Figure 3.3 Permeameter (ACS laboratories)

$$k_{gas} = \frac{2000 \cdot BP \cdot \mu \cdot Q \cdot L}{[(P_1 \cdot 0.06805 + BP)^2 - (BP)^2] \cdot A} \quad (3.1)$$

$$k_{gas(actual)} = k_{gas} \; x \; 0.9716 \quad (3.2)$$

Where:

k	is the permeability, (md)
q	is the flow rate, (cc/sec)
μ	is the gas viscosity, (cp)
L	is the sample length, (cm)
A	is the cross-sectional area, (cm^2)
P_1	is the inlet pressure, (psig)
BP	is the barometric pressure, (atm)
0.06805	is conversion factor for psi to atmosphere

0.9716 is conversion factor for the expansion of air due to saturation with water vapor in the bubble tube.

3.3.3 Thickening time

Cement slurry was filled in mold, removed the excess gently and made a smooth upper surface. Immediately after leveling the slurry, transferred mold and base-plate to the Vicat apparatus (use needle diameter 1 mm.). Lower the needle until it contacts to surface of slurry, pause in that position 1 second – 2 second. Then recorded value after released the needle 30 second. Repeated the test after 15 minute to the same specimen and wait until needle penetrated to 25 millimeter, recorded initial setting time (total time after mixed). Final setting time was the total time after cement sample were mixed until needle penetrated only 0.5 millimeter into specimen.

Figure 3.4 Vicat apparatus

3.3.4 Density

Cement slurry was filled in pressurized fluid density beam balance apparatus below the edge of cup. Placed the lid on the cup and check valve must be in open position. Then used the pressurized pump filled slurry up the cup untill it was full. Placed the instrument on the knife edge and slided balanced weight right or left until the beam was balanced. Read and recorded specific grarity of cement slurry at the balance point.

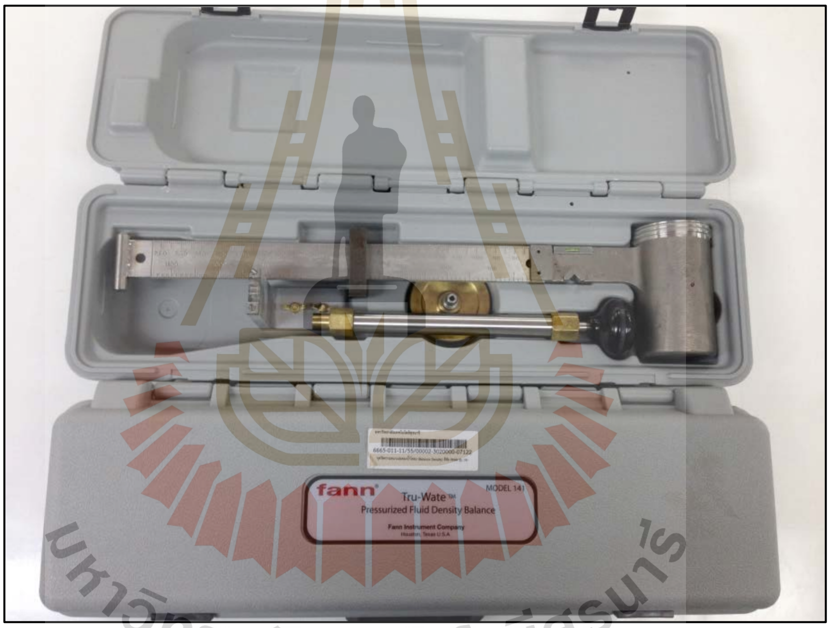


Figure 3.5 Pressurized fluid density balance (Fann model 141)

3.3.5 Filtration

Cement slurry was full filled in the test cell of the apparatus. Applied pressure 100 psi to the cell and recorded the filtrate volume for 30 second, 1 minute, 2 minute, 5 minute, 7 minute, 10 minute, 15 minute, 25 minute, and 30 minute. If bleed out occurred before 30 minute, recorded the filtrate volume and time at which the bleed out occurred and calculated the ISO fluid loss by using equation 3.3.

$$\text{ISO fluid loss} = V_t \frac{10.944}{\sqrt{t}} \qquad (3.3)$$

If the test could be run entire 30 min without bleed out, measured the filtrate volume, doubled the value and reported it as the fluid loss value.

Where:

V_t is the volume of filtrate collected at the time of the bleed out, (milliliters)

t is the time of bleed out, (minutes)

Figure 3.6 Filtration test apparatus (Fann model 821)

3.3.6 Viscosity

Cement slurry was filled in the viscometer cup below the edge of cup. The apparent viscosity (μ_a), plastic viscosity (μ_p) and yield point (γ_p) were calculated from 300 and 600 rpm reading according to equation 3.4, 3.5 and 3.6 respectively.

$$\mu_a = \phi_{600}/2 \tag{3.4}$$

$$\mu_p = \phi_{600}/\phi_{300} \tag{3.5}$$

$$\gamma_p = \phi_{300}/\mu_p \tag{3.6}$$

Where:

ϕ_{600} is the viscosity dial reading at 600 rpm

ϕ_{300} is the viscosity dial reading at 300 rpm

μ_a is the apparent viscosity, (cp)

μ_p is the plastic viscosity, (cp)

γ_p is the Yield point, (lbf/100 ft^2)

The shear stress (τ) is determined as a function of the shear rate (γ). The shear stress and shear rate of cement slurry samples were then calculated by using equation 3.7 and 3.8 respectively

$$\tau = 0.01066\phi_i N \tag{3.7}$$

$$\gamma = 1.703\text{rpm} \tag{3.8}$$

Where:

τ is the shear stress, (lb$_f$/ft^2)

γ is the shear rate, (sec^{-1})

ϕ_i is the viscometer dial reading

N is the range extension factor of the torque spring of the VG meter

Rpm is the rotational speed, (round per minutes)

The power law model's parameters in the term of behavior index (n) and consistency (k) can be calculated from viscometer reading by equation 3.9 and 3.10.

$$n = 3.322\log(\phi_{600}/\phi_{300}) \tag{3.9}$$

$$k = 510\phi_{300}/511^{n} \tag{3.10}$$

Where:

n is the flow behavior index

k is the fluid consistency index

ϕ_{600} is the viscosity dial reading at 600 rpm

ϕ_{300} is the viscosity dial reading at 300 rpm

Figure 3.7 Viscometer (Fann model 35SA)

CHAPTER IV

RESULT AND DISCUSSIONS

4.1 Introduction

This chapter describes the results of laboratory experiments which were used to determinate chemical properties, physical properties, rheological properties, and cost comparison between cement and cement mixed with RHA that used in petroleum well drilling respectively.

4.2 Chemical properties of RHA

4.2.1 Test methods

RHA samples were analyzed their chemical compound and the results by X-ray fluorescence spectrometer are shown in Table 4.1 and Figure 4.1.

Table 4.1 Chemical compound of RHA samples of this study

Chemical compound	Percent by weight (%)
SiO_2	96.72
CaO	1.33
K_2O	1.92
Others	0.03
Total	100

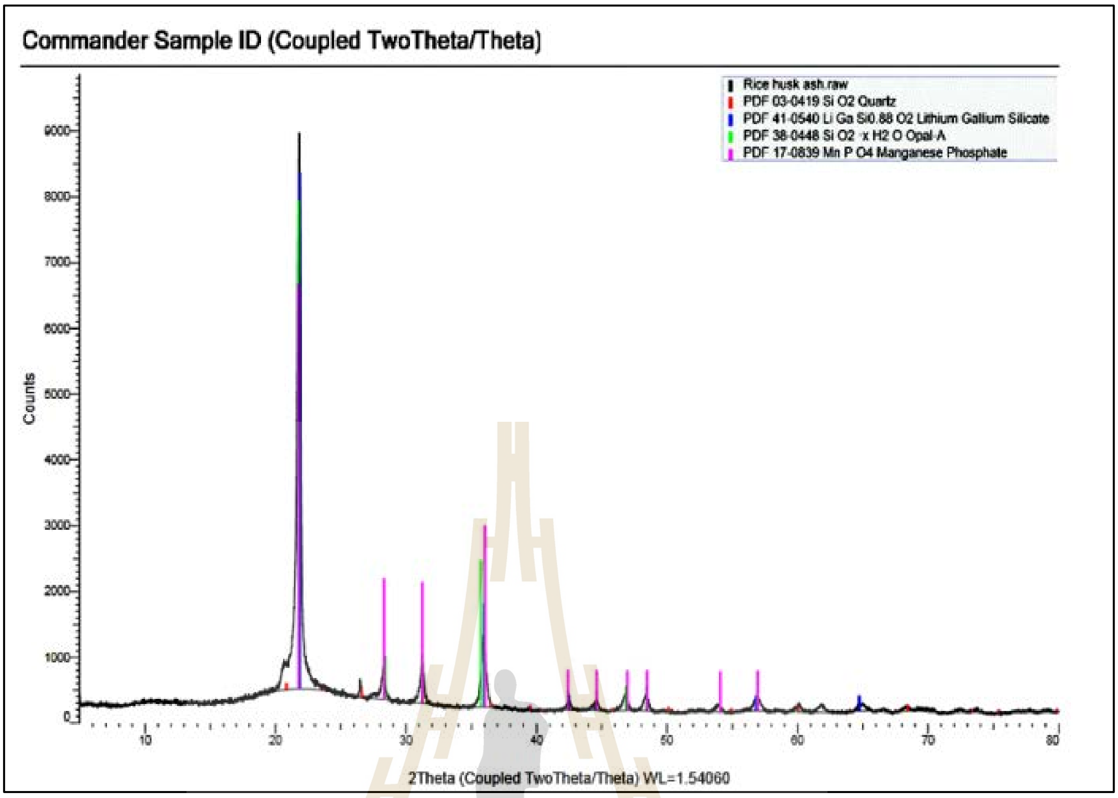


Figure 4.1 XRD result of RHA sample

Results from XRD analysis indicated that RHA samples were mainly composed of SiO_2 (96.72 wt%) and some minor compounds as CaO (1.33 wt%), K_2O (1.92 wt%) and the rest are other compounds. High SiO_2 is benefit for increasing the cement strength.

4.3 Physical properties of cement samples

These tests were conducted to determine strength, permeability and thickening time of cement and RHA cement. Cement slurry samples were mixed by water/binder ratio at 0.5 and RHA was added to replace cement at 0 wt%, 5 wt%, 10 wt%, 15 wt% and 20 wt% respectively.

4.3.1 Strength

Strength of samples was measured by the compress machines. Tested samples were cured at 2 temperatures as 25 degree Celsius and 80 degree Celsius, for 3, 7, 14, 28, 56 days, and were dried before taken to the test instrument. For each condition, there were 3 concrete samples had been tested. Results of the compressive strength test at various temperatures and curing time are depicted in Table 4.2 to Table 4.11 and Figure 4.2 to Figure 4.13, respectively.

Table 4.2 Strength of cement at 25°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	0 wt%	80.94	80.46	81.41	80.94
7	0 wt%	111.67	110.83	114.52	112.34
14	0 wt%	115.92	114.48	116.52	115.64
28	0 wt%	119.15	117.74	115.56	117.48
56	0 wt%	124.50	126.34	120.06	123.63

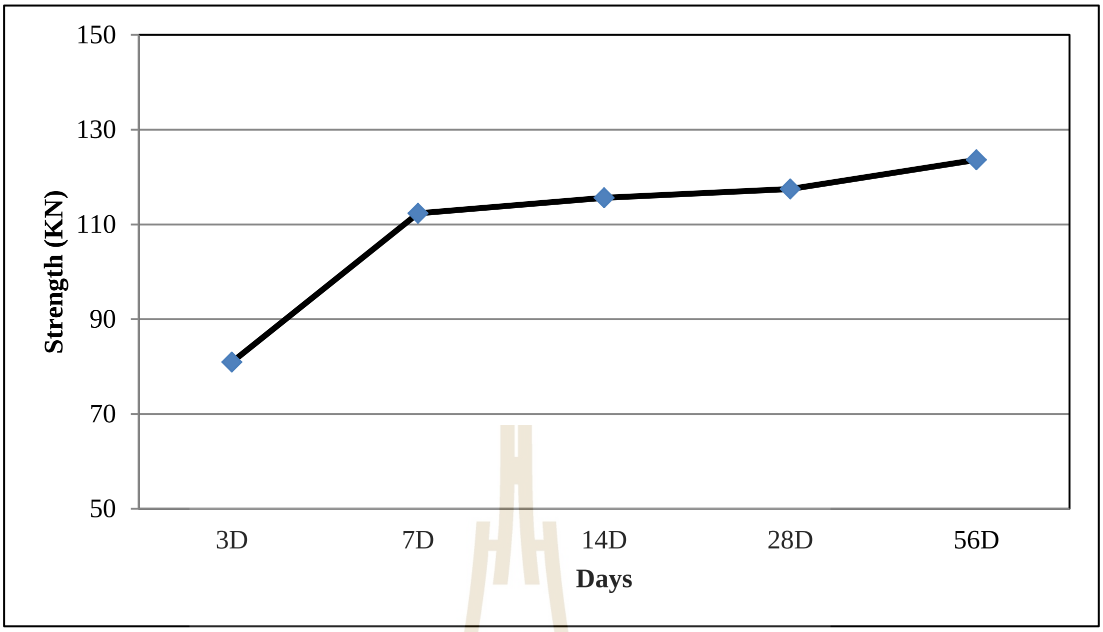


Figure 4.2 Strength of cement at 25°C curing temperature

Table 4.3 Strength of cement with RHA 5 wt% at 25°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	5 wt%	8134	82.17	79.73	81.08
7	5 wt%	108.90	113.56	110.87	111.11
14	5 wt%	123.46	127.83	124.37	125.22
28	5 wt%	135.12	134.43	138.94	136.16
56	5 wt%	143.68	147.64	144.27	145.20

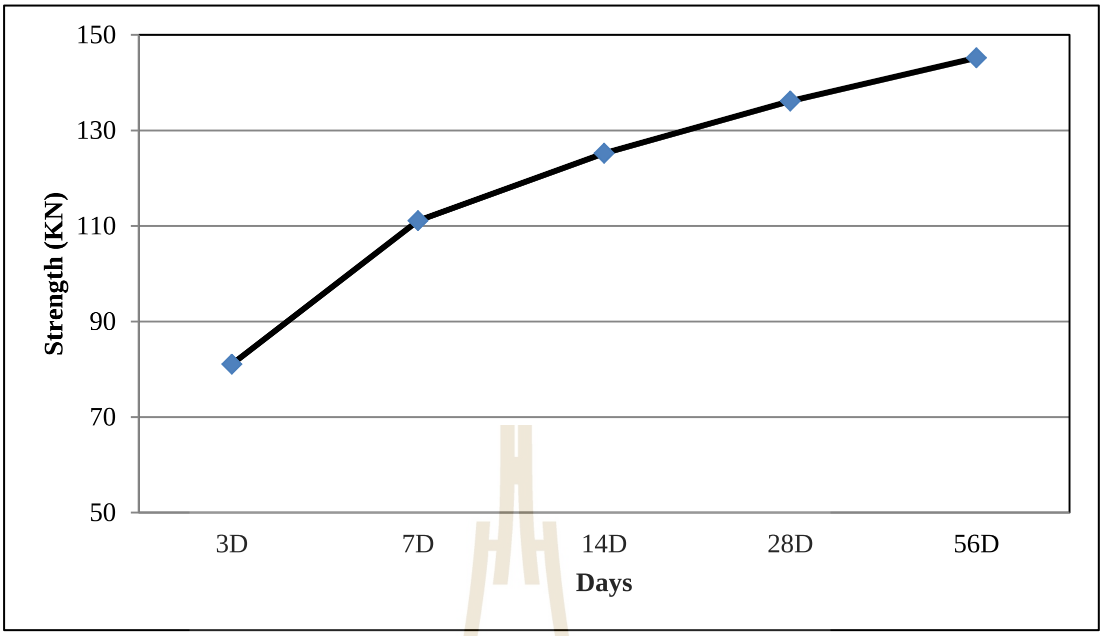


Figure 4.3 Strength of cement with RHA 5 wt% at 25°C curing temperature

Table 4.4 Strength of cement with RHA 10 wt% at 25°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	10 wt %	80.06	80.90	83.10	81.35
7	10 wt %	135.00	136.30	138.49	136.60
14	10 wt %	144.22	148.74	146.17	146.38
28	10 wt %	154.47	155.36	154.45	154.76
56	10 wt %	173.20	176.40	175.60	175.07

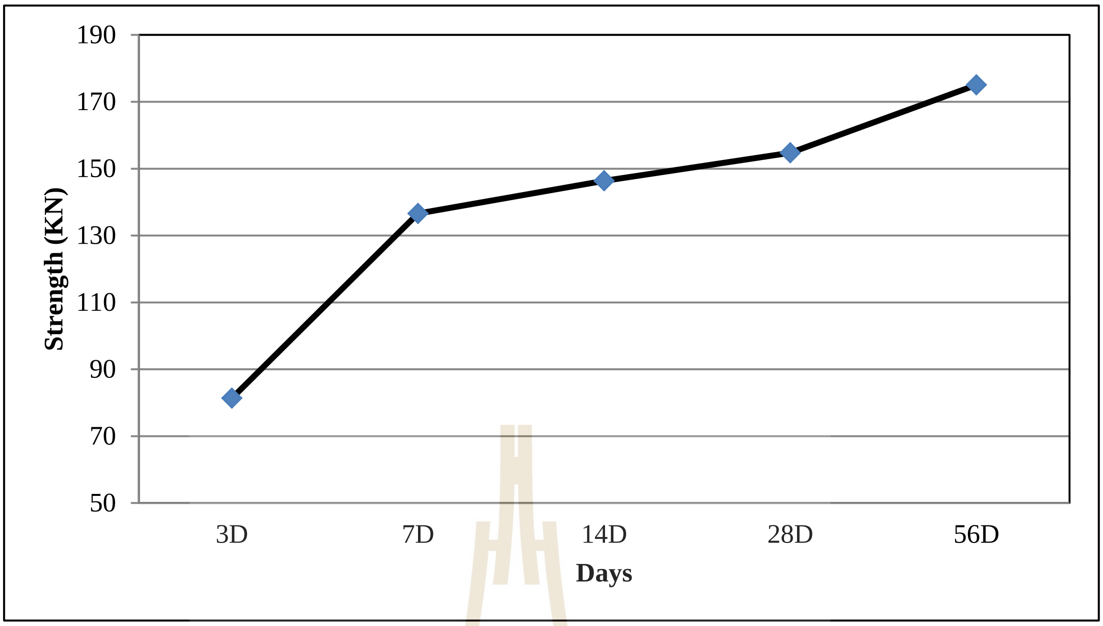


Figure 4.4 Strength of cement with RHA 10 wt% at 25°C curing temperature

Table 4.5 Strength of cement with RHA 15 wt% at 25°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	15 wt%	82.30	78.11	85.25	81.80
7	15 wt%	140.19	143.23	143.10	142.17
14	15 wt%	149.64	153.51	145.85	149.67
28	15 wt%	155.80	159.11	157.30	157.40
56	15 wt%	178.94	180.40	176.70	178.68

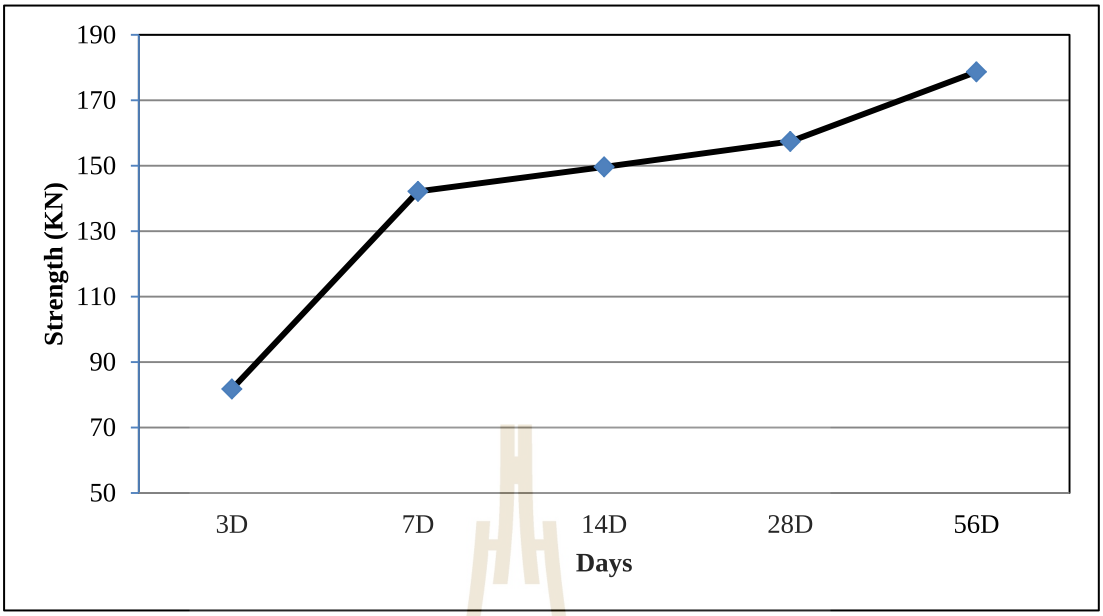


Figure 4.5 Strength of cement with RHA 15 wt% at 25°C curing temperature

Table 4.6 Strength of cement with RHA 20 wt% at 25°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	20 wt%	83.16	82.38	83.76	83.10
7	20 wt%	145.30	146.60	145.84	145.85
14	20 wt%	154.28	151.94	150.50	152.24
28	20 wt%	160.81	157.10	158.43	158.78
56	20 wt%	179.51	183.40	185.10	182.67

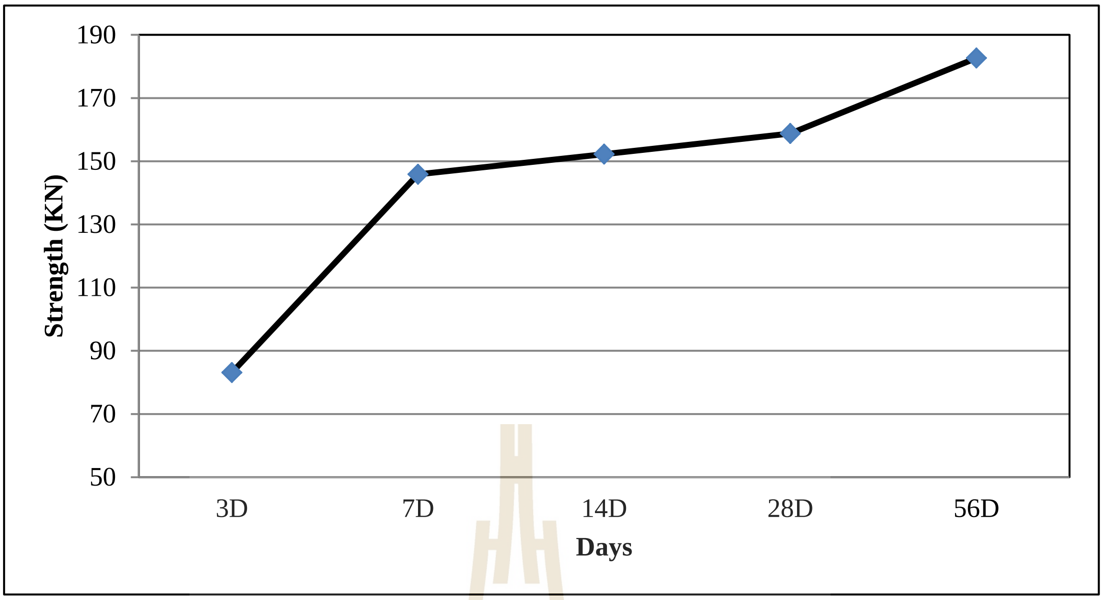


Figure 4.6 Strength of cement with RHA 20 wt% at 25°C curing temperature

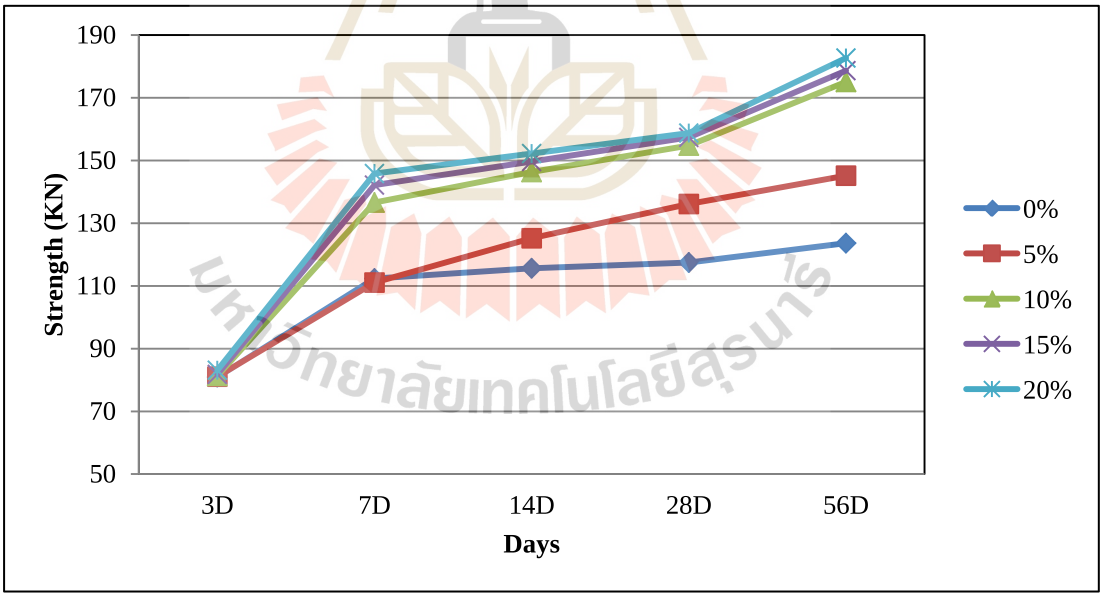


Figure 4.7 Average strength of cement and cement with RHA at various weight percent and 25°C curing temperature

Table 4.7 Strength of cement at 80°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	0 wt%	81.32	80.23	81.12	80.89
7	0 wt%	113.21	111.79	109.37	111.46
14	0 wt%	114.56	116.43	115.45	115.48
28	0 wt%	118.85	117.22	119.54	118.54
56	0 wt%	127.74	125.35	122.22	124.10

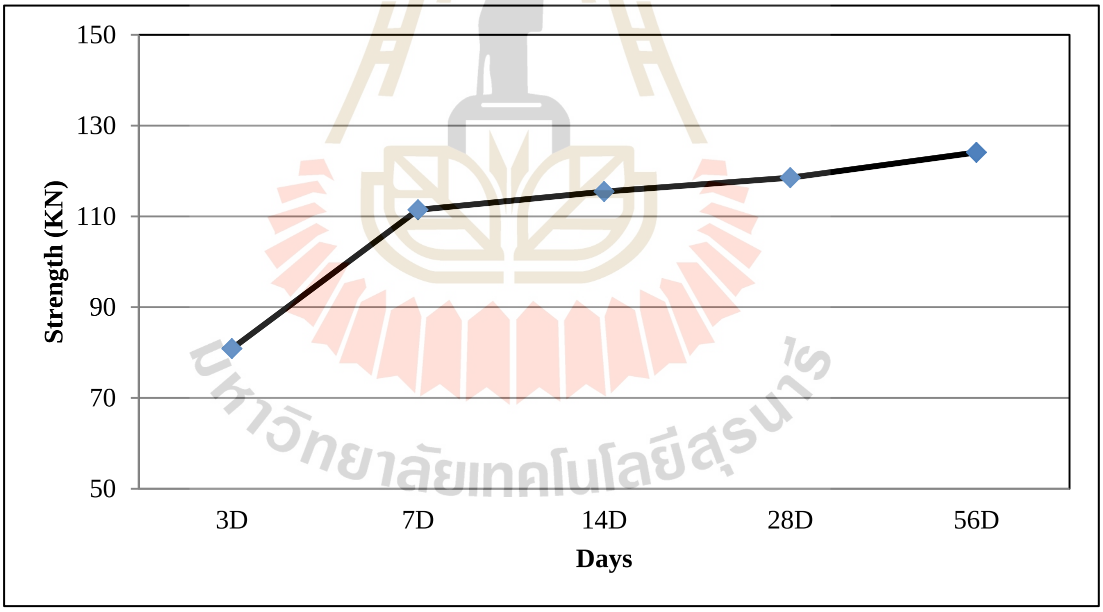


Figure 4.8 Strength of cement at 80°C curing temperature

Table 4.8 Strength of cement with RHA 5 wt% at 80°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	5 wt%	82.30	80.10	81.00	81.13
7	5 wt%	113.29	115.64	110.01	112.98
14	5 wt%	122.48	126.94	124.33	124.58
28	5 wt%	133.25	135.61	138.00	135.62
56	5 wt%	145.77	145.46	142.96	144.73

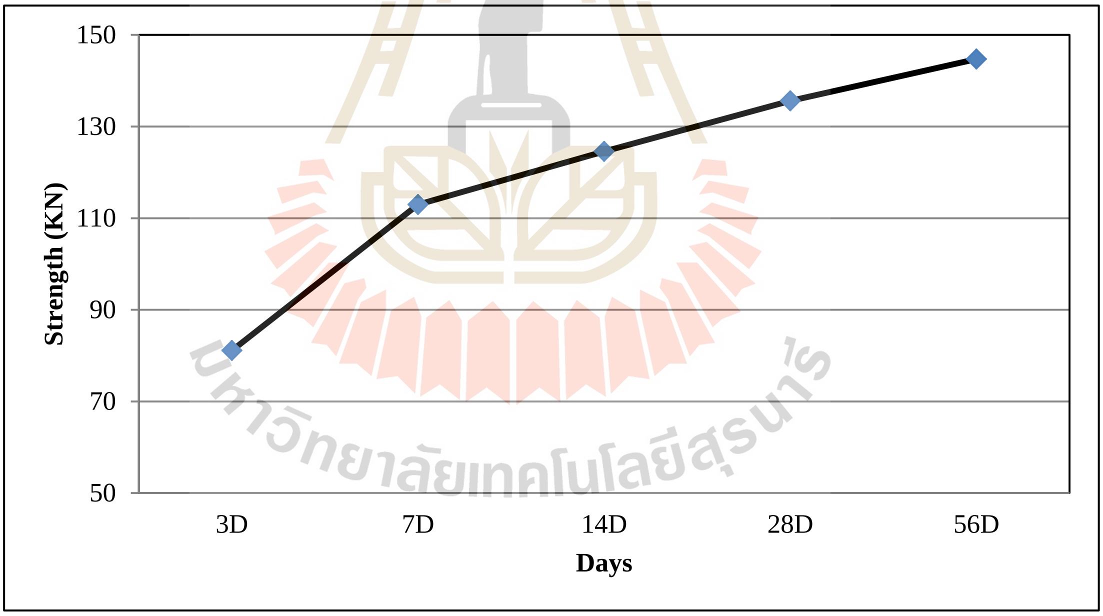


Figure 4.9 Strength of cement with RHA 5 wt% at 80°C curing temperature

Table 4.9 Strength of cement with RHA 10 wt% at 80°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	10 wt%	81.00	80.20	83.10	81.43
7	10 wt%	136.26	133.022	136.15	135.21
14	10 wt%	144.00	146.74	148.03	146.26
28	10 wt%	155.89	155.36	155.07	155.44
56	10 wt%	177.91	175.36	174.84	176.04

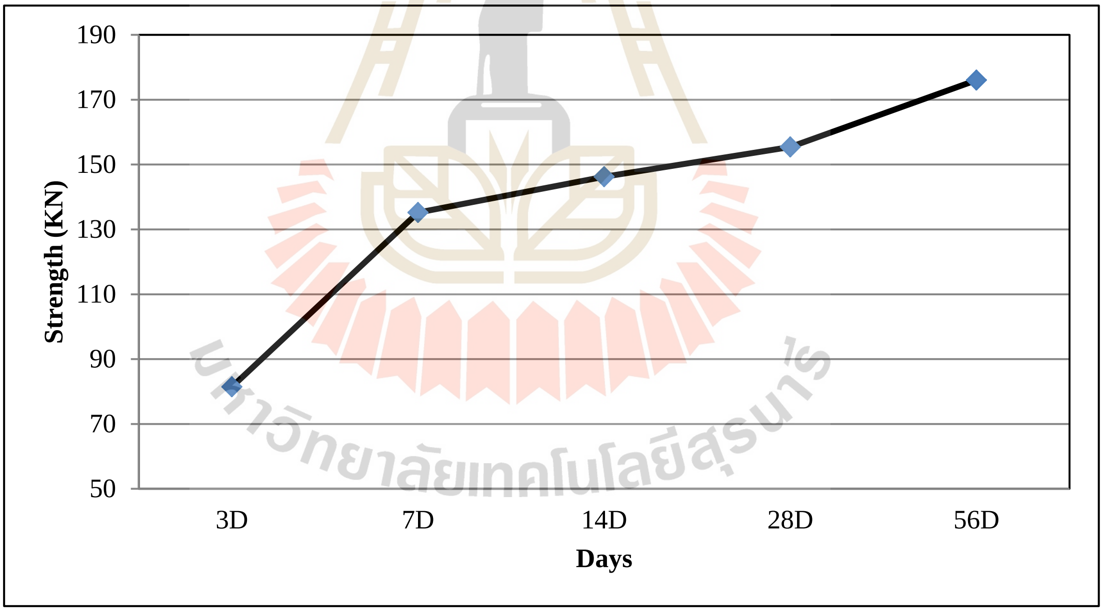


Figure 4.10 Strength of cement with RHA 10 wt% at 80°C curing temperature

Table 4.10 Strength of cement with RHA 15 wt% at 80°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	15 wt%	81.11	80.48	83.44	81.68
7	15 wt%	143.27	142.85	140.49	142.20
14	15 wt%	148.44	147.69	150.00	148.71
28	15 wt%	158.57	158.33	158.40	158.43
56	15 wt%	178.00	178.88	179.36	178.75

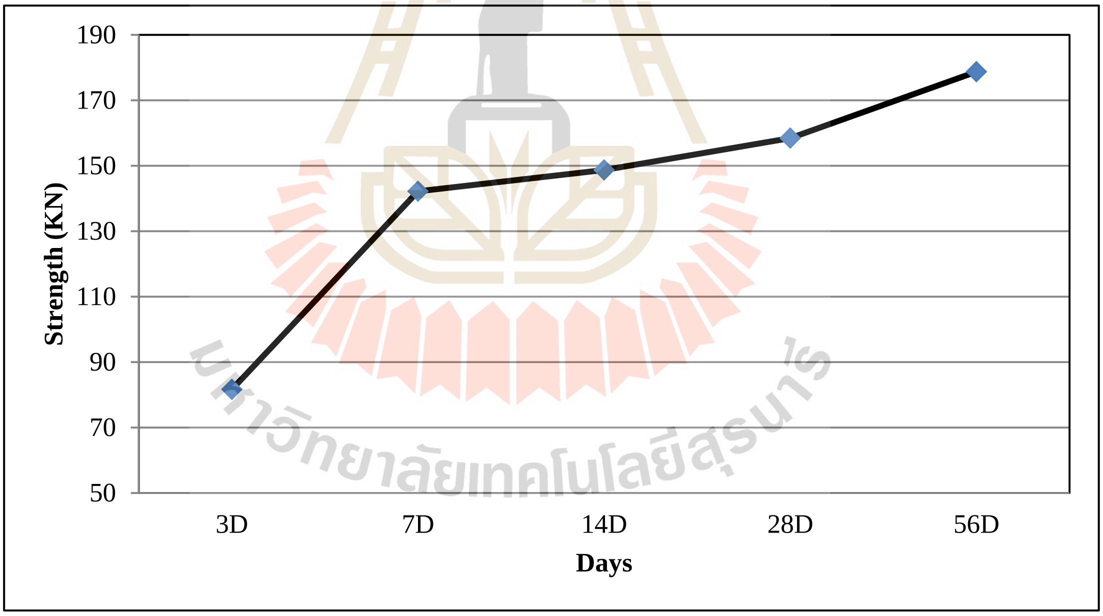


Figure 4.11 Strength of cement with RHA 15 wt% at 80°C curing temperature

Table 4.11 Strength of cement with RHA 20 wt% at 80°C curing temperature

DAYS	RHA	STRENGTH (KN)			AVERAGE
		Test No.1	Test No.2	Test No.3	
3	20 wt%	84.00	80.67	83.41	82.69
7	20 wt%	144.78	144.68	145.07	144.84
14	20 wt%	151.66	150.95	151.74	151.45
28	20 wt%	159.98	160.01	158.85	159.61
56	20 wt%	181.81	180.93	184.15	182.30

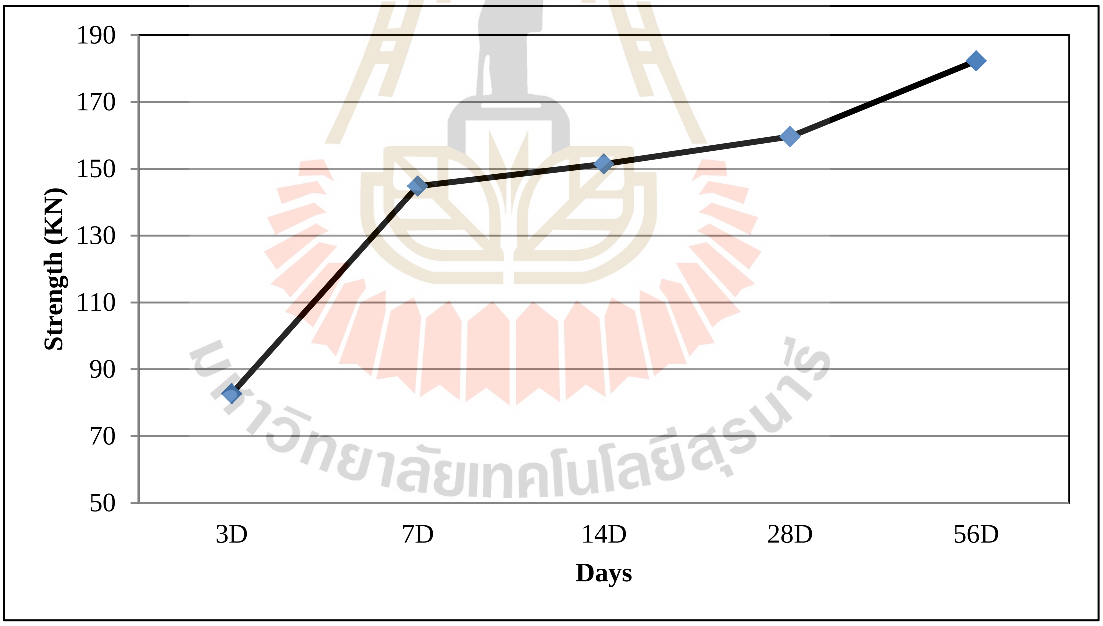


Figure 4.12 Strength of cement with RHA 20 wt% at 80°C curing temperature

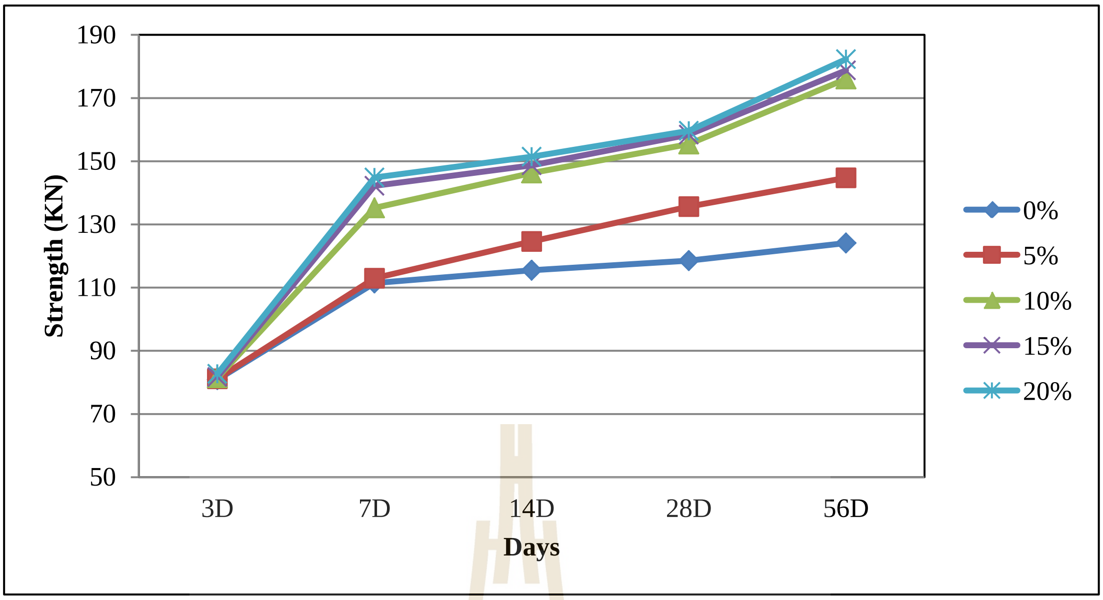


Figure 4.13 Average strength of cement and cement with RHA at various weight percent and 80°C curing temperature

Results of the compressive strength test in this study indicated that concretes which were replaced by RHA 20 wt% had the highest compressive strength compared to other weight percent. This may cause from the RHA which was used in this study had very high SiO_2 content (96.72 wt%). This might decrease the binder/space ratio which is resulted in decreasing the pore space of the concrete, and consequently result in increasing the strength of the concrete specimens. Not only the very high SiO_2 content, but also the higher curing temperature at 25°C and 80°C, especially at 80°C, may also cause the pozzolanic reaction take place to the concrete specimens earlier than those of previous work.

It can also noticeable that the longer curing time concrete, the higher coment compressive strength.

4.3.2 Permeability

Set cement specimens were measured their permeability by using Permeameter apparatus at the Geotechnology Laboratory, Suranaree University of Technology. The cement specimen was measured its permeability at various pressure level and results of the measurement are listed in Table 4.12 to Table 4.16, respectively.

Table 4.12 Permeability of cement without RHA

Dimension of specimen Length 4.93 centimeter Diameter 3.84 centimeter

Cross-sectional area 11.59 square centimeter

Pc (psi)	P_1 (psi)	P_1 (atm)	P_b (atm)	Viscosity (cp)	V (cm^3)	Time (sec)	Q (cm^3/sec)	K_gas (millidarcy)
560	40.05	2.72	1	0.0173	5	14.25	0.0351	0.392
550	30.10	2.05	1	0.0173	5	17.03	0.294	0.509
540	20.09	1.37	1	0.0173	5	27.22	0.184	0.574
530	10.05	0.68	1	0.0173	2	24.31	0.082	0.644
3100	40.05	2.72	1	0.0173	5	15.31	0.327	0.365
3000	30.08	2.05	1	0.0173	5	22.22	0.225	0.390
3200	20.07	1.37	1	0.0173	5	37.03	0.135	0.422
3200	10.04	0.68	1	0.0173	2	30.53	0.066	0.514

Table 4.13 Permeability of cement with RHA 5 wt%

Dimension of specimen Length 4.92 centimeter Diameter 3.79 centimeter

Cross-sectional 11.28 square centimeter

Pc (psi)	**P_1 (psi)**	**P_1 (atm)**	**P_b (atm)**	**Viscosity (cp)**	**V (cm^3)**	**Time (sec)**	**Q (cm^3/sec)**	**K_gas (millidarcy)**
620	40.04	2.72	1	0.0173	5	18.47	0.271	0.314
610	30.06	2.04	1	0.0173	5	24.71	0.202	0.360
600	20.09	1.37	1	0.0173	5	38.91	0.129	0.411
580	10.04	0.68	1	0.0173	2	33.15	0.060	0.485
3100	40.02	2.72	1	0.0173	5	26.13	0.191	0.219
3000	30.05	2.04	1	0.0173	5	34.66	0.144	0.257
3000	20.06	1.36	1	0.0173	2	21.28	0.094	0.302
2900	10.06	0.68	1	0.0173	2	46.19	0.043	0.347

Table 4.14 Permeability of cement with RHA 10 wt%

Dimension of specimen Length 4.88 centimeter Diameter 3.79 centimeter

Cross-sectional 11.31 square centimeter

Pc (psi)	P_1 (psi)	P_1 (atm)	P_b (atm)	Viscosity (cp)	V (cm^3)	Time (sec)	Q (cm^3/sec)	K_gas (millidarcy)
510	40.09	2.73	1	0.0173	5	21.40	0.234	0.264
500	30.21	2.06	1	0.0173	2	12.16	0.164	0.288
610	20.37	1.39	1	0.0173	2	19.78	0.101	0.314
580	10.05	0.68	1	0.0173	2	41.41	0.048	0.384
3000	40.04	2.72	1	0.0173	5	30.54	0.164	0.185
2900	30.12	2.05	1	0.0173	5	43.34	0.115	0.203
3000	20.03	1.36	1	0.0173	2	28.66	0.070	0.222
2900	10.10	0.69	1	0.0173	2	61.50	0.033	0.257

Table 4.15 Permeability of cement with RHA 15 wt%

Dimension of specimen Length 4.91 centimeter Diameter 3.80 centimeter

Cross-sectional 11.34 square centimeter

Pc (psi)	P_1 (psi)	P_1 (atm)	P_b (atm)	Viscosity (cp)	V (cm^3)	Time (sec)	Q (cm^3/sec)	K_gas (millidarcy)
570	40.08	2.73	1	0.0173	5	42.59	0.117	0.133
550	30.00	2.04	1	0.0173	5	57.28	0.087	0.155
530	20.04	1.36	1	0.0173	2	37.31	0.054	0.171
510	10.03	0.68	1	0.0173	2	73.41	0.027	0.218
3200	40.06	2.73	1	0.0173	5	50.97	0.098	0.111
3000	30.03	2.04	1	0.0173	5	71.16	0.080	0.124
2950	20.05	1.36	1	0.0173	2	46.00	0.043	0.139
3200	10.10	0.69	1	0.0173	2	101.10	0.020	0.157

Table 4.16 Permeability of cement with RHA 20 wt%

Dimension of specimen Length 5.00 centimeter Diameter 3.80 centimeter

Cross-sectional 11.34 square centimeter

Pc (psi)	P_1 (psi)	P_1 (atm)	P_b (atm)	Viscosity (cp)	V (cm^3)	Time (sec)	Q (cm^3/sec)	K_gas (millidarcy)
650	40.07	2.73	1	0.0173	5	66.19	0.076	0.087
620	30.05	2.04	1	0.0173	5	63.01	0.079	0.143
610	20.04	1.36	1	0.0173	2	41.04	0.049	0.158
600	10.05	0.68	1	0.0173	2	88.58	0.023	0.183
3100	40.01	2.72	1	0.0173	5	56.07	0.089	0.103
3100	30.11	2.05	1	0.0173	5	78.28	0.064	0.115
3000	20.15	1.37	1	0.0173	2	50.60	0.040	0.127
3000	10.10	0.69	1	0.0173	2	111.21	0.018	0.145

It was found that the permeability was reversely proportional to the weight percentages of RHA; it was decreased when the weight percentage of RHA was increased. This is because RHA used in this study contained very high SiO2 content, and then result in decreasing the pore space of the concrete specimen when using in larger amount.

4.3.3 Thickening time

Thickening (setting) time of concrete specimen both with RHA and without RHA were measured by the Vicat apparatus at the Civil Engineering Laboratory, Suranaree University of Technology. Results of the measurement are shown in Table 4.17 and Figure 4.14 respectively.

Table 4.17 Final setting time of cement with and without RHA

Sample	Final setting time (min)
RHA 0 wt%	330
RHA 5 wt%	315
RHA 10 wt%	270
RHA 15 wt%	270
RHA 20 wt%	270

Results from thickening time measurement indicated that the thickening time of cement slurry was reverse proportional to the RHA weight percent replacement since higher silica content can help cement slurry form and bond its internal structure easier.

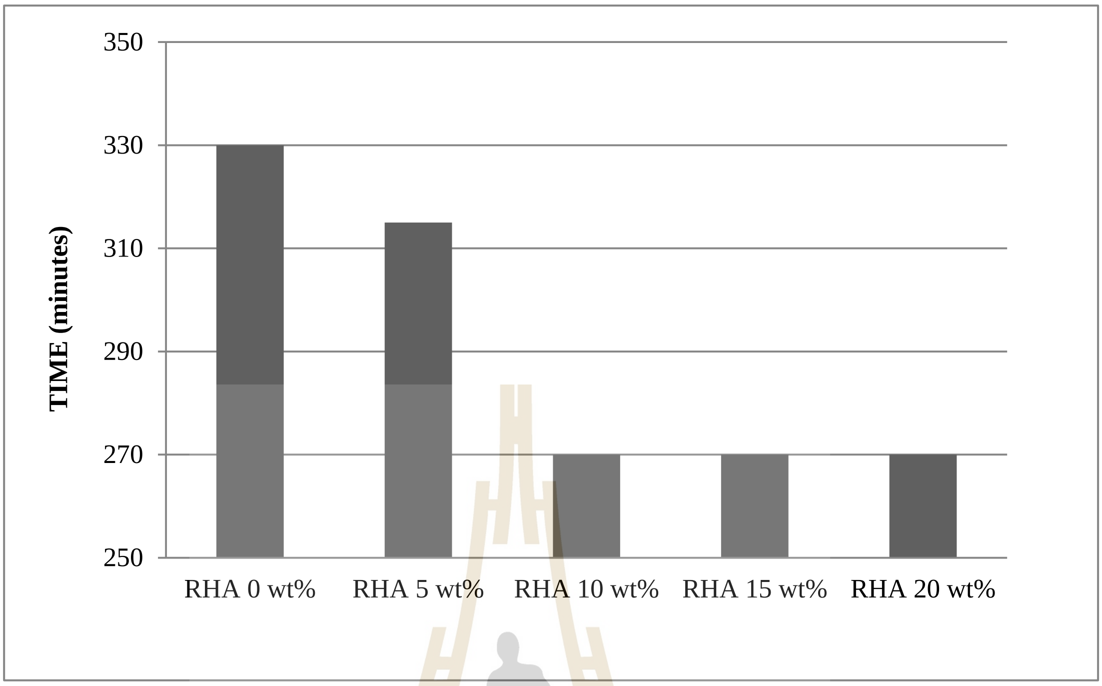


Figure 4.14 Final setting time of cement with and without RHA

4.4 Rheological properties

Rheological properties measurement in this study included density, volume, viscosity, gel strength, and filtrate loss measurement of cement with and without RHA. Cement slurry samples were mixed by water/binder ratio at 0.5 and added RHA instead of cement at 0 wt%, 5 wt%, 10 wt%, 15 wt%, and 20 wt% respectively.

4.4.1 Density

Density of the cement slurry samples in this study was measured by Pressurized fluid density balance. Results of measurement are shown in Table 4.18 and Figure 4.15 to Figure 4.17.

Table 4.18 Density of cement and cement with RHA

Sample	Fluid density			Pressure gradient	Volume (cc)
	g/cc	lb/gal	lb/cu ft	lb/sq in/1000ft	
RHA 0%	1.835	15.30	114.50	795.00	985
RHA 5%	1.810	15.00	110.00	780.00	995
RHA 10%	1.800	15.00	110.00	780.00	1005
RHA 15%	1.760	14.70	108.00	765.00	1015
RHA 20%	1.750	14.70	108.00	765.00	1025

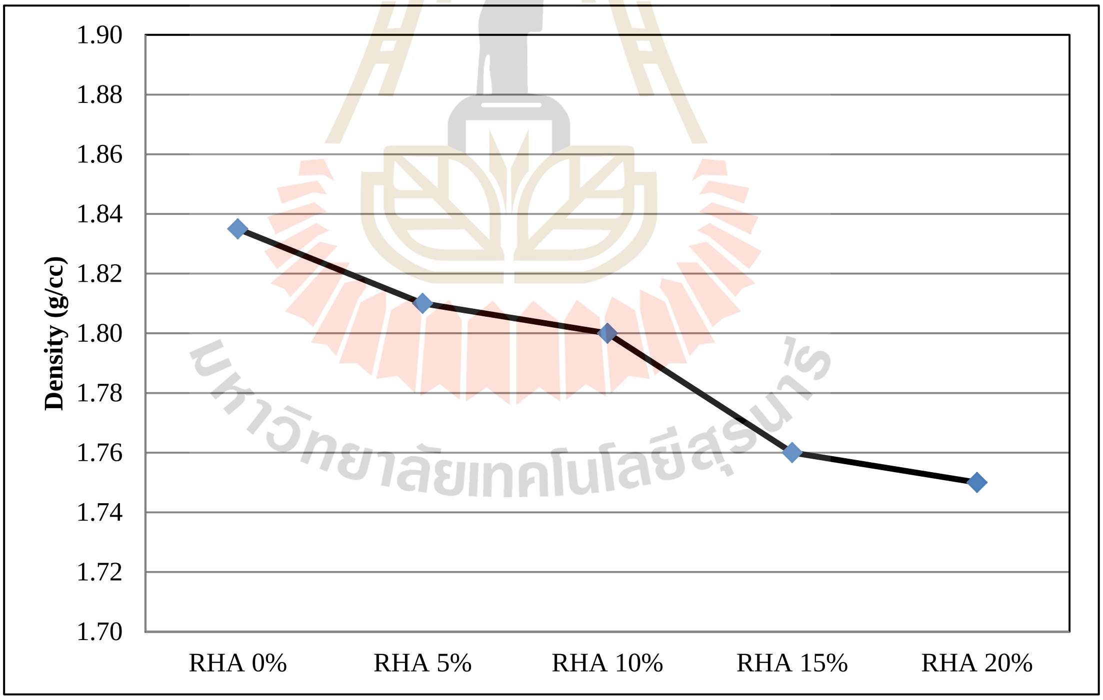


Figure 4.15 Density of cement and cement with RHA in grams/cubic centimeter

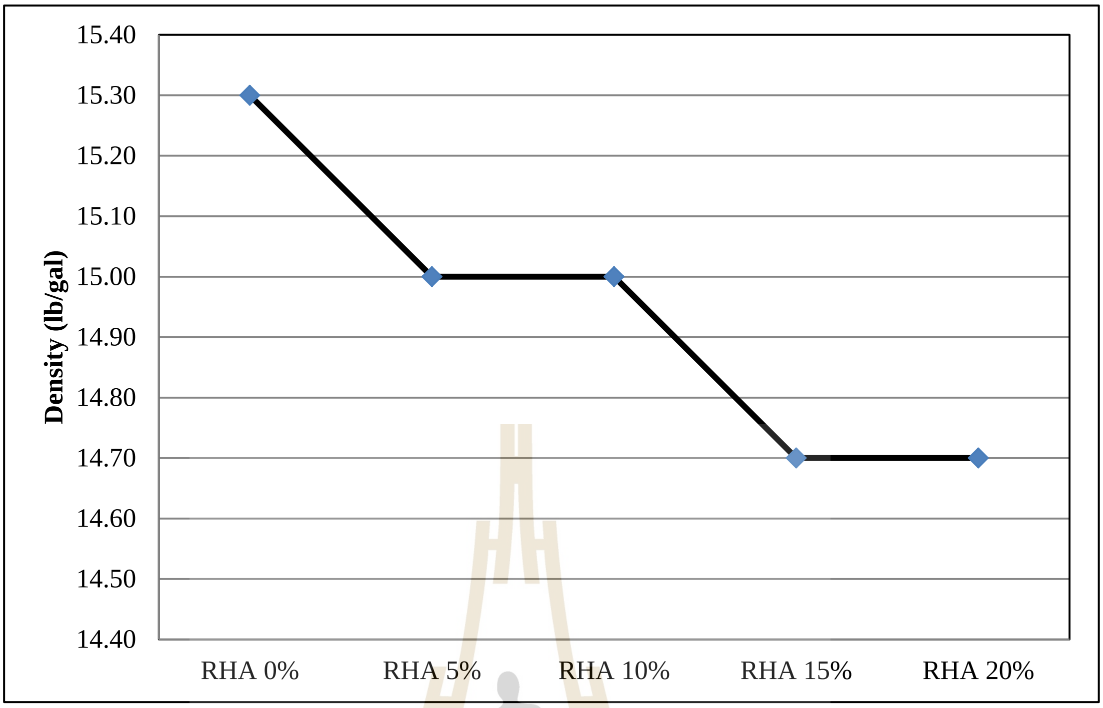


Figure 4.16 Density of cement and cement with RHA in pound/gallon

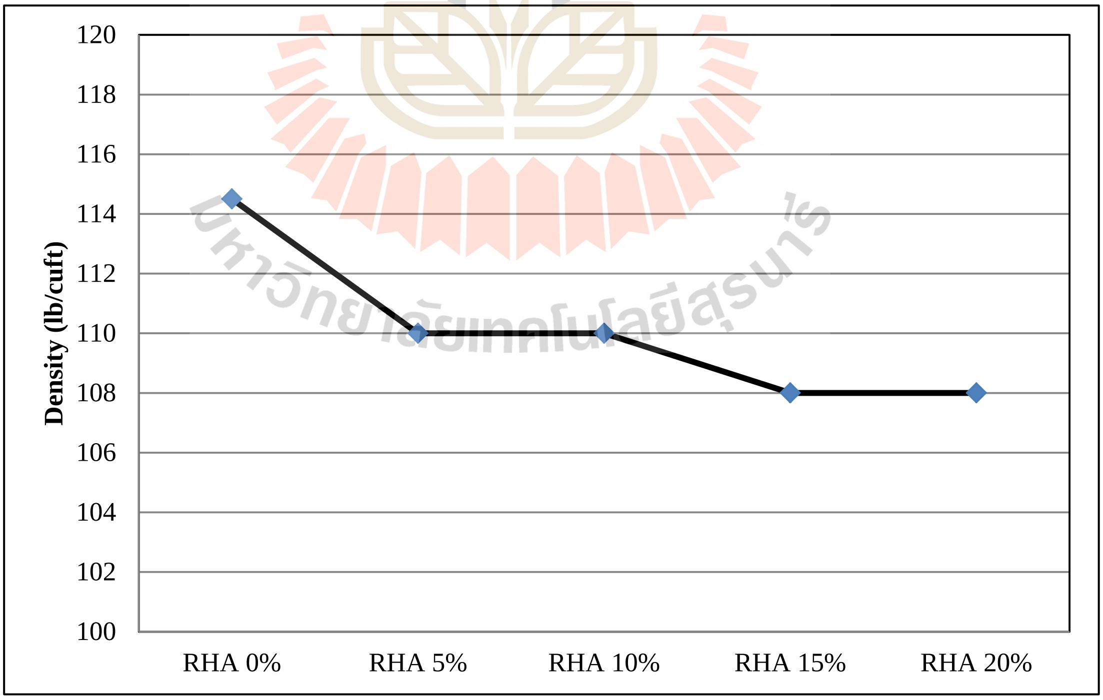


Figure 4.17 Density of cement and cement with RHA in pounds/cubic foot

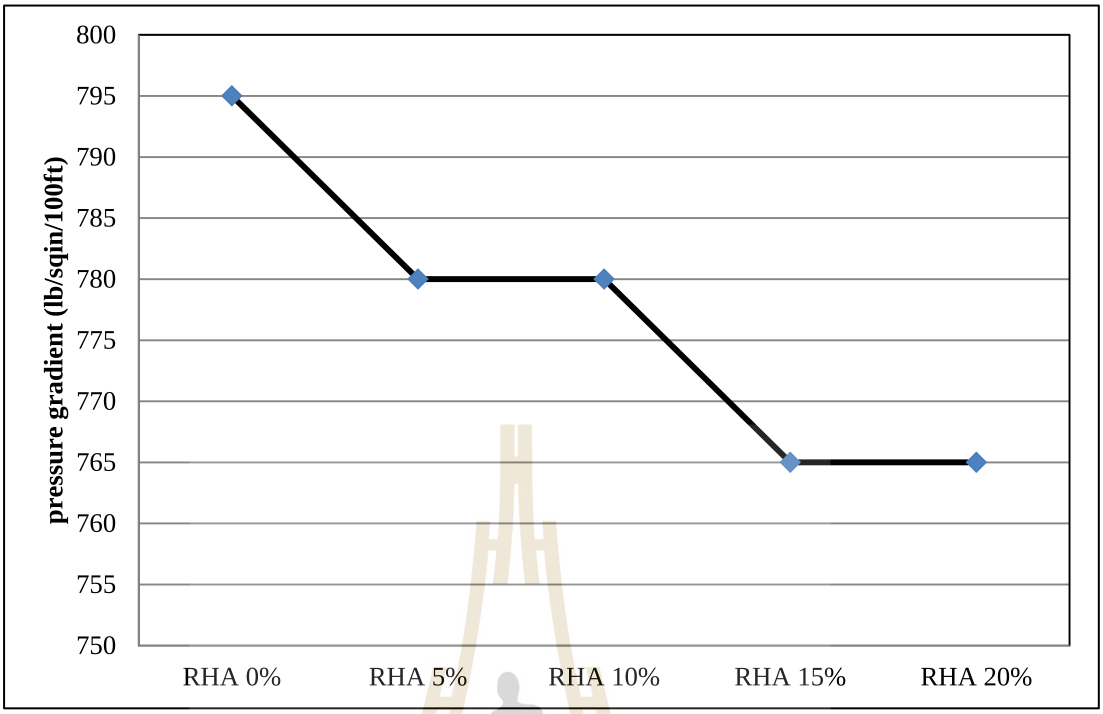


Figure 4.18 Pressure gradients of cement and cement with RHA in pounds per square inch per 1000 foot

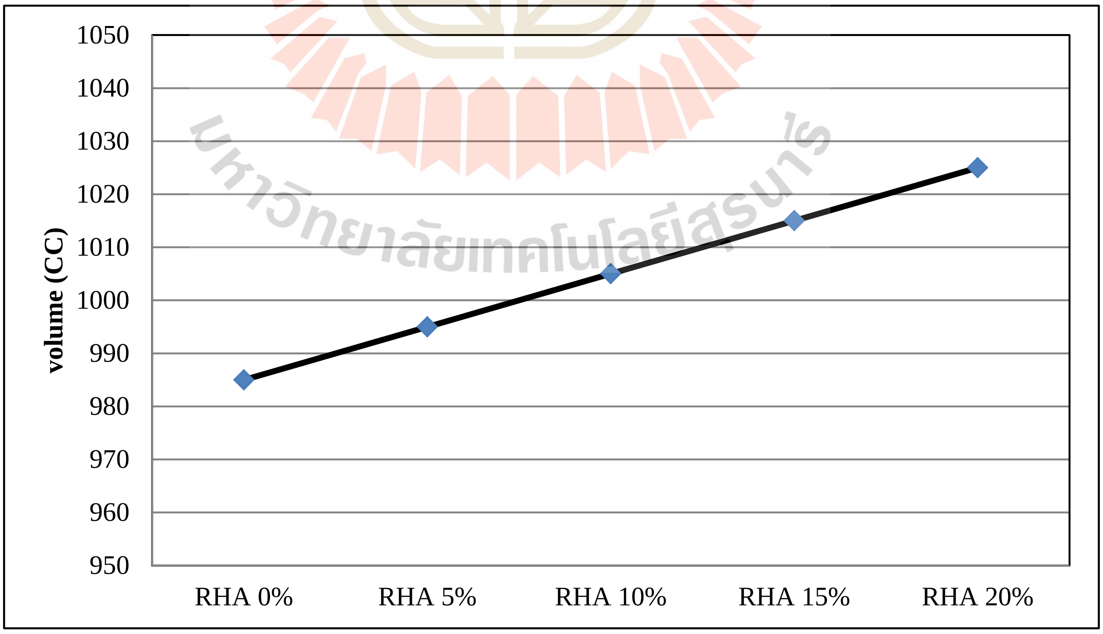


Figure 4.19 Volume of cement and cement with RHA

Results revealed that density of cement slurry sample was reverse proportional to the weight percent of RHA replacement (Figure 4.15 to Figure 4.18). It was also found that the volume of cement slurry samples increased with the increasing of RHA weight percent (Figure 4.19). Therefore, this might be supported that RHA can be used as an extender additive for cement.

4.4.2 Viscosity and gel strength

Viscosity and gel strength of the cement slurry samples were measured by viscometer as described in section 3.3.6. Results of the measurement are depicted in Table 4.19 to Table 4.24 and Figure 4.20 to Figure 4.24, respectively.

Table 4.19 Viscosity of cement slurry samples with and without RHA

Sample	Plastic viscosity (cp)	Apparent viscosity (cp)	Yield point (lb/100 ft^2)
RHA 0 wt%	89	124.5	71
RHA 5 wt%	27	46.5	39
RHA 10 wt%	55	66	35
RHA 15 wt%	70	85	30
RHA 20 wt%	N/A	N/A-	N/A-

Results from cement slurry samples viscosity measurement indicated that viscosity of the RHA cement slurry was direct proportional to the amount of RHA (Table 4.19).

Table 4.20 Shear stress and shear rate of cement slurry samples without RHA

RPM	**Average read**	**Shear rate** **γ (sec^{-1})**	**Shear stress** **τ (lb_f/ft^2)**
600	249	1021.8	0.531
300	161	510.9	0.343
200	131	340.6	0.279
100	92	170.3	0.196
6	19	10.2	0.041
3	15	5.1	0.032

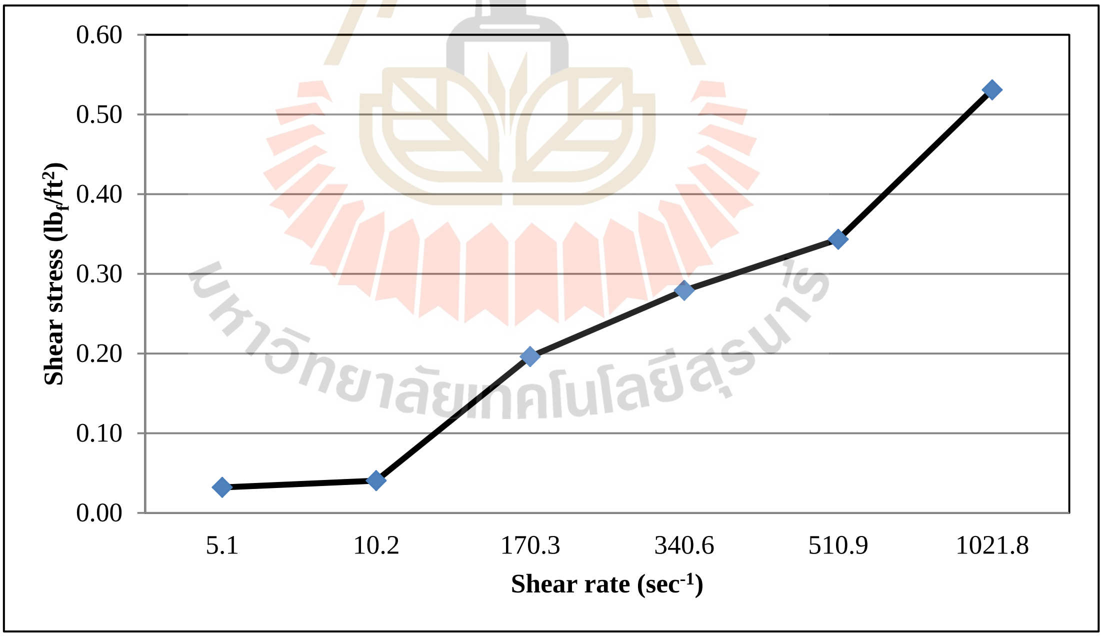


Figure 4.20 Shear stress and shear rate of cement

Table 4.21 Shear stress and shear rate of cement with RHA 5 wt%

RPM	**Average read**	**Shear rate** γ **(sec^{-1})**	**Shear stress** τ **(lb$_f$/ft^2)**
600	104	1021.8	0.222
300	69	510.9	0.147
200	58	340.6	0.124
100	42	170.3	0.090
6	12	10.2	0.026
3	9	5.1	0.019

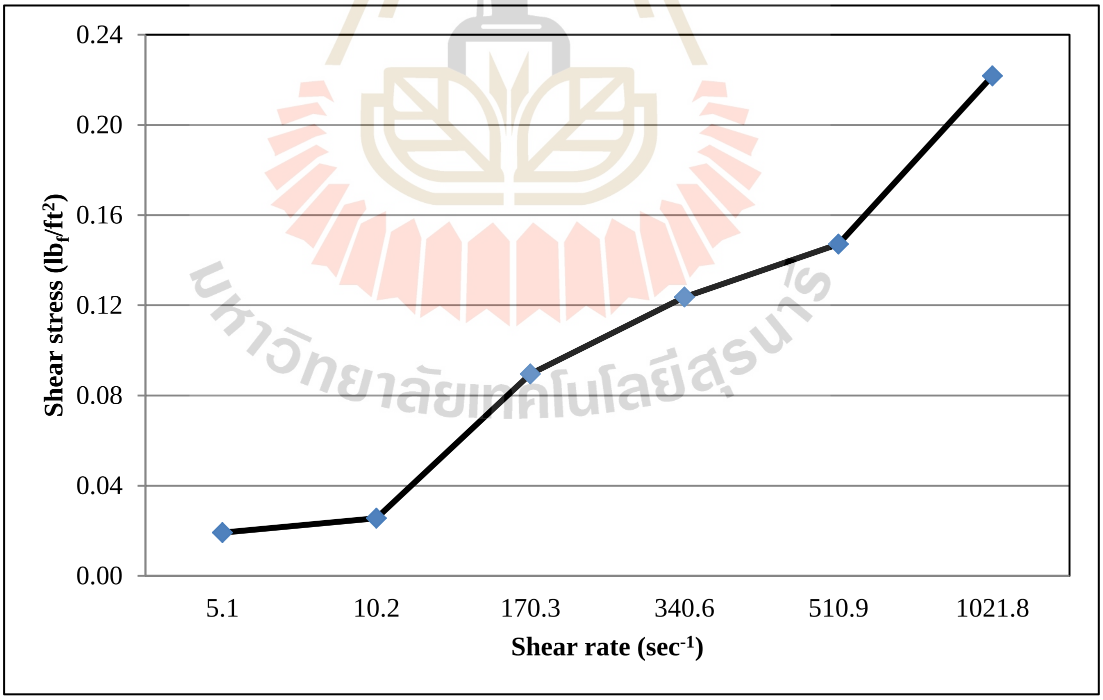


Figure 4.21 Shear stress and shear rate of cement with RHA 5 wt%

Table 4.22 Shear stress and shear rate of cement with RHA 10 wt%

RPM	**Average read**	**Shear rate** **γ (sec^{-1})**	**Shear stress** **τ (lb$_f$/ft^2)**
600	130	1021.8	0.277
300	77	510.9	0.164
200	59	340.6	0.126
100	41	170.3	0.087
6	11	10.2	0.023
3	8	5.1	0.017

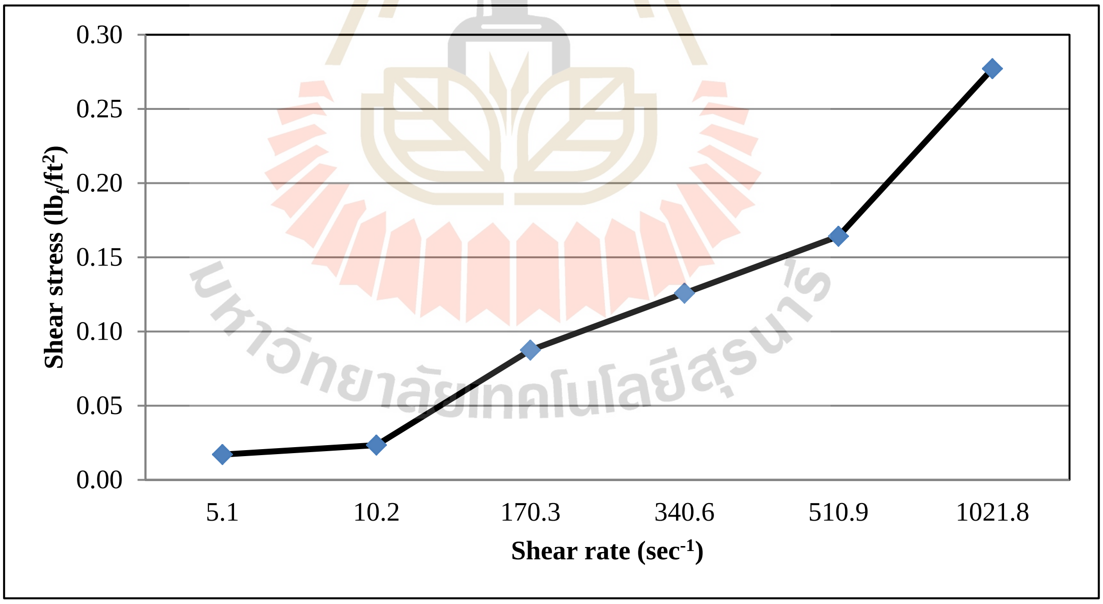


Figure 4.22 Shear stress and shear rate of cement with RHA 10 wt%

Table 4.23 Shear stress and shear rate of cement with RHA 15 wt%

RPM	**Average read**	**Shear rate** γ **(sec^{-1})**	**Shear stress** τ **(lb$_f$/ft^2)**
600	160	1021.8	0.341
300	103	510.9	0.220
200	74	340.6	0.158
100	52	170.3	0.111
6	12	10.2	0.026
3	9	5.1	0.019

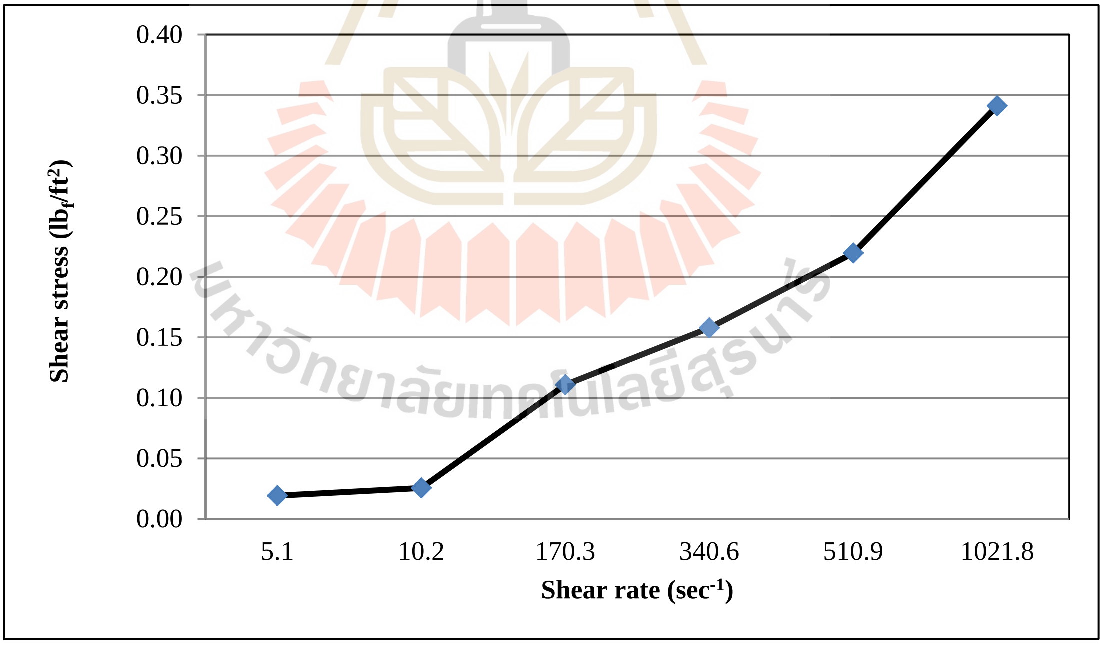


Figure 4.23 Shear stress and shear rate of cement with RHA 15 wt%

Table 4.24 Shear stress and shear rate of cement with RHA 20 wt%

RPM	**Average read**	**Shear rate γ (sec^{-1})**	**Shear stress τ (lb$_f$/ft^2)**
600	-	1021.8	-
300	194	510.9	0.414
200	140	340.6	0.298
100	78	170.3	0.166
6	15	10.2	0.032
3	10	5.1	0.021

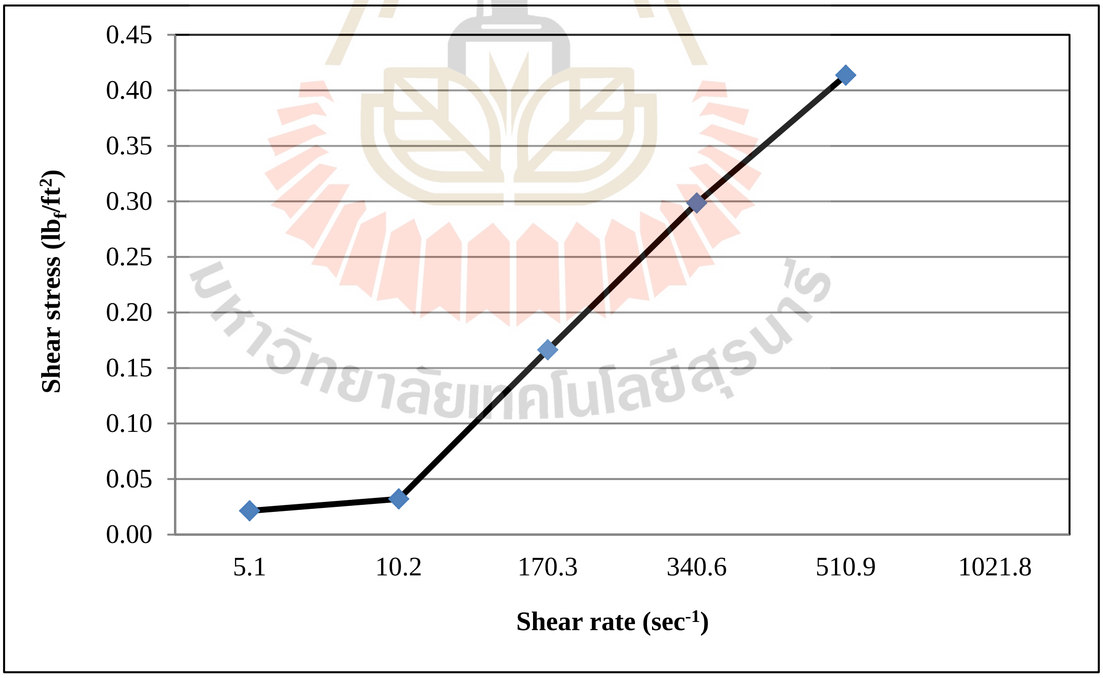


Figure 4.24 Shear stress and shear rate of cement with RHA 20 wt%

4.4.3 Filtrate

Cement slurry samples filtrate loss volume were measured by filtration test apparatus as described in section 3.3.5. Results of measurements are shown in Table 4.25 and Figure 4.25.

Table 4.25 Fluid loss in 30 minute at 1,000 psi

Sample	Fluid loss (milliliter)
RHA 0 wt%	75
RHA 5 wt%	57
RHA 10 wt%	79
RHA 15 wt%	93
RHA 20 wt%	112

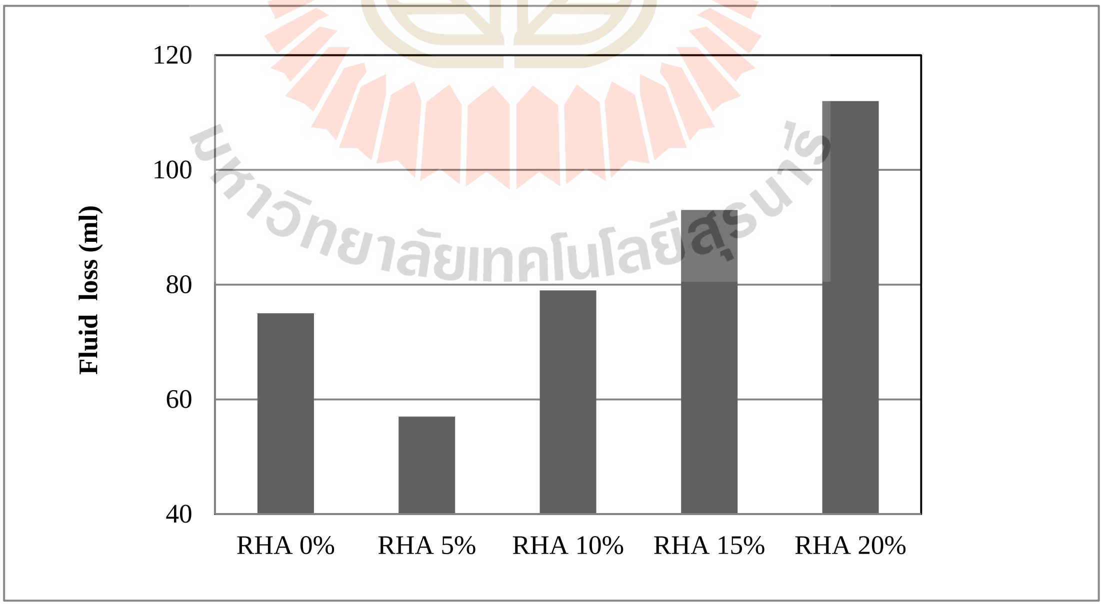


Figure 4.25 Fluid loss in 30 minute at 1,000 psi

Results of filtrate volume measurement in this study indicated that the filtrate volume was direct proportional to the RHA amount. This is may cause by the water which is absorbed by RHA is squeezed out easily under the pressurized condition during the RHA cement is still in a plastic phase. However, the amount of filtrate volume may be decreased when the pozzolanic reaction take place occurs and cement is well set.

4.5 Economic consideration

This section presents the cost comparison between using cement with and without RHA. To scope the cost comparison, oil well cement cost of TPI Polene (4 Thai Bath/kg) and RHA cost of Thai Naronk Kut Chick Ltd. (0.5 Thai Bath/kilogram) were used. It was also assumed that 10,000 kilogram of cement was used in this calculation.

In term of economics consideration, results from calculation showed in Table 4.26 indicate that total cost can be decreased if RHA is used larger. This is because the cost of RHA is about 8 times cheaper than cement.

Table 4.26 Cost comparison between cement with and without RHA

Sample	Cement (Kilograms)	RHA (Kilograms)	Cost of cement (Baht)	Cost of RHA (Baht)	Total cost (Baht)
RHA 0%	1,0000	0	40,000	0.0	40,000.00
RHA 5%	9,500	5	38,000	2.5	38,002.50
RHA 10%	9,000	10	36,000	5.0	36,605.00
RHA 15%	8,500	15	34,000	7.5	34,907.50
RHA 20%	8,000	20	32,000	10.0	33,210.00

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Based on experiments and measurements of this study, RHA chemical compound, physical properties, including compressive strength and thickening time, and rheological properties, e.g. density, viscosity, filtrate volume of cement with and without RHA were concluded and summarized as follows.

5.1.1 RHA chemical compound

Result from XRD analysis indicated the RHA sample used in this study was mainly composed of SiO_2 (96.72 wt%), CaO (1.33 wt%), K_2O (1.92 wt%) and Other compounds (0.03 wt%) respectively.

5.1.2 Physical properties

Results from compressive strength measurement indicated that the compressive strength of set cement was direct proportional to the amount of RHA weight percent. This is because the pore space within set cement specimen is replaced by silicon dioxide and result in pore space decreasing. Moreover, since RHA samples used in this study is composed of very high SiO_2 content, this may cause the quicker pozzolanic reaction and also result in compressive strength enhancing.

This study also found that permeability of set cement specimens was reverse proportional to the amount of RHA weight percent. The reason might be the same as in the compressive strength that silicon dioxide is filled up the pore space within set cement specimen and make it smaller.

Thickening time of RHA cement was also reverse proportional to the amount of RHA weight percent. This is because the pozzolanic reaction can be taken place earlier since RHA samples used in this study contain very high SiO_2 which can help cement slurry form and bond it internal structure easier.

5.1.3 Rheological properties

It was found from the study that density of RHA cement was direct proportional to the amount of RHA in plastic phase. This is because RHA can absorb larger amount of water when the RHA amount is increased and make it is lighten.

However, the larger amount of RHA can cause the cement slurry more viscous because the hydration and pozzolanic reaction can be taken place more easily and quickly.

Filtrate volume measurement on RHA cement slurry indicated that in the plastic phase filtrate volume of RHA cement slurry was direct proportional to the amount of RHA. This is because the water that is hydrated with RHA can be squeezed out easily under the pressurized condition during the RHA cement slurry is still in the plastic phase. This is one of the disadvantage points of using RHA mixing with cement if the filtrate volume is taken into account.

Results from the study also indicated that the volume of cement slurry could be increased by adding RHA as replacement material for cement.

5.1.4 Economics consideration

Since RHA is the agricultural byproduct that is low cost, abundant, and easily available, and it can be used as an oil well cement additive to increase the cement slurry volume, enhance the compressive strength of set cement, etc., RHA can be used as a replacement material for cement economically.

Results from experiments, measurements, and analysis on the physical properties, rheological properties, and economics consideration are summarized and shown in Table 5.1.

Therefore, RHA can be used as oil well cement to increase the cement slurry volume, reduce viscosity and save cement cost. However, there are some disadvantages points of using RHA mixing with cement, especially during cement is in plastic phase as it reduces density of the cement slurry, and increase the filtrate volume of cement slurry which are unfavorable conditions during the cement replacement process in petroleum well drilling.

However, when considered all tests and measurement results, RHA can be used as an oil well additive if it is used to replace cement at 10 %wt. This is because reduction of density and filtrate loss volume of the cement slurry are not much different from those of cement without RHA, whereas the compressive strength is higher.

5.2 Recommendations

For the future study, RHA samples should be collected from various places to study the effect of SiO_2 content and the physical properties and rheological properties of set cement and cement slurry. RHA more than 20 wt% should be tested further to compare and select appropriate RHA weight percent which will be used to replace the oil well cement.

Tested temperature should be elevated to higher than 80°C to study the effect of temperature to the pozzolanic effect since the real bottom hole temperature of petroleum well is normally higher than 80°C.

Table 5.1 Summary of physical properties, rheological properties and cost comparison of cement with and without RHA

Samples	Physical properties				Rheological properties			Cost different (%)
	Strength Average 56 days (KN)		Permeability (millidarcy)	Thickening time (min)	Density (ppg)	Viscosity [PV] (centipoises)	Filtrate loss (ml)	
	25°C	80°C						
RHA 0 wt%	123.63	124.10	0.644	330	15.30	89	75	0.00
RHA 5 wt%	145.20	144.73	0.485	315	15.00	27	57	-5.00
RHA 10 wt%	175.07	176.04	0.384	270	15.00	55	79	-8.50
RHA 15 wt%	178.68	178.75	0.218	270	14.70	70	93	-12.73
RHA 20 wt%	182.67	182.30	0.183	315	14.70	N/A	112	-16.98

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APPENDIX A

EXPERIMENTAL DATA

Pressurized Fluid Balance data and parameter for all tested

Table A1 Density for RHA 0 wt% (No.1)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	31	1.835	15.30	114.5	795.0
2	31	1.835	15.30	114.5	795.0

Table A2 Density for RHA 0 wt% (No.2)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	30	1.830	15.25	114.0	790.0
2	30	1.830	15.25	114.0	790.0

Table A3 Density for RHA 0 wt% (No.3)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	32	1.835	15.30	114.5	795.0
2	32	1.835	15.30	114.5	795.0

Table A4 Density for RHA 5 wt% (No.1)

Pressurized Fluid Balance					
Test	Temp	Fluid Density			Pressure Gradient
	(°C)	g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	29	1.810	15.00	110.0	780.0
2	29	1.810	15.00	110.0	780.0

Table A5 Density for RHA 5 wt% (No.2)

Pressurized Fluid Balance					
Test	Temp	Fluid Density			Pressure Gradient
	(°C)	g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	30	1.810	15.00	110.0	780.0
2	30	1.810	15.00	110.0	780.0

Table A6 Density for RHA 5 wt% (No.3)

Pressurized Fluid Balance					
Test	Temp	Fluid Density			Pressure Gradient
	(°C)	g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	29	1.810	15.00	110.0	780.0
2	29	1.810	15.00	110.0	780.0

Table A7 Density for RHA 10 wt% (No.1)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	30	1.80	15.00	110	780
2	30	1.80	15.00	110	780

Table A8 Density for RHA 10 wt% (No.2)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	30	1.80	15.00	110	780
2	30	1.80	15.00	110	780

Table A9 Density for RHA 10 wt% (No.3)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	30	1.80	15.00	110	780
2	30	1.80	15.00	110	780

Table A10 Density for RHA 15 wt% (No.1)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	31	1.760	14.70	108.00	765
2	31	1.760	14.70	108.00	765

Table A11 Density for RHA 15 wt% (No.2)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	30	1.760	14.70	108.00	765
2	30	1.760	14.70	108.00	765

Table A12 Density for RHA 15 wt% (No.3)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	31	1.760	14.70	108.00	765
2	31	1.760	14.70	108.00	765

Table A13 Density for RHA 20 wt% (No.1)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	31	1.750	14.70	108.00	765
2	31	1.750	14.70	108.00	765

Table A14 Density for RHA 20 wt% (No.2)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	31	1.750	14.70	108.00	765
2	31	1.750	14.70	108.00	765

Table A15 Density for RHA 20 wt% (No.3)

Pressurized Fluid Balance					
Test	Temp (°C)	Fluid Density			Pressure Gradient
		g/cc	lb/gal	lb/ft^3	lb/sq in/100 ft
1	31	1.750	14.70	108.00	765
2	31	1.750	14.70	108.00	765

Fan viscometer data and parameter for all tested

Table A16 Viscosity &Gel strength for RHA 0 wt%

RPM	Reading#1	Reading#2	Reading#3	Reading#4	Average Reading	γ (sec^{-1})	τ (lb_f/ft^2)
600	249	247	250	249	249	1021.8	0.531
300	162	159	162	162	161	510.9	0.343
200	130	133	130	129	131	340.6	0.279
100	91	91	95	89	92	170.3	0.196
6	19	19	18	20	19	10.2	0.041
3	15	15	13	16	15	5.1	0.032
PV		89					
AV		124.5					
YP		71					
Gel_{10s}		15					
Gel_{10min}		13					

Table A17 Viscosity &Gel strength for RHA 5 wt%

RPM	Reading#1	Reading#2	Reading#3	Reading#4	Average Reading	γ (sec^{-1})	τ (lb_f/ft^2)
600	99	95	110	110	104	1021.8	0.222
300	66	65	73	72	69	510.9	0.147
200	55	55	61	61	58	340.6	0.124
100	40	41	43	45	42	170.3	0.090
6	11	12	11	14	12	10.2	0.026
3	9	9	9	10	9	5.1	0.019
PV		27					
AV		46.5					
YP		39					
Gel_{10s}		9					
Gel_{10min}		9					

Table A18 Viscosity &Gel strength for RHA 10 wt%

RPM	Reading#1	Reading#2	Reading#3	Reading#4	Average Reading	γ (sec^{-1})	τ (lb_f/ft^2)
600	132	125	130	133	130	1021.8	0.277
300	77	75	78	78	77	510.9	0.164
200	63	59	54	60	59	340.6	0.126
100	42	42	37	42	41	170.3	0.087
6	12	13	9	11	11	10.2	0.023
3	9	9	7	8	8	5.1	0.017
PV		55					
AV		66					
YP		22					
Gel_{10s}		9					
Gel_{10min}		7					

Table A19 Viscosity &Gel strength for RHA 15 wt%

RPM	Reading#1	Reading#2	Reading#3	Reading#4	Average Reading	γ (sec^{-1})	τ (lb_f/ft^2)
600	170	165	165	140	160	1021.8	0.341
300	100	97	125	90	103	510.9	0.220
200	60	76	85	75	74	340.6	0.158
100	50	50	60	48	52	170.3	0.111
6	12	12	10	12	12	10.2	0.026
3	9	9	7	9	9	5.1	0.019
PV		70					
AV		85					
YP		30					
Gel_{10s}		9					
Gel_{10min}		7					

Table A20 Viscosity &Gel strength for RHA 20 wt%

RPM	Reading#1	Reading#2	Reading#3	Reading#4	Average Reading	γ (sec^{-1})	τ (lb_f/ft^2)
600	N/A	N/A	N/A	N/A	N/A	1021.8	-
300	195	195	195	190	194	510.9	0.414
200	140	139	140	140	140	340.6	0.298
100	82	82	64	82	78	170.3	0.166
6	16	15	15	15	15	10.2	0.032
3	8	8	6	7	10	5.1	0.021
PV		N/A					
AV		N/A					
YP		N/A					
Gel_{10s}		8					
Gel_{10min}		6					

Fluid loss data for all tested

Table A21 Fluid loss for RHA 0 wt% (No.1)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	20		
#2	1	30	75	2.15
#3	2	75		

Table A22 Fluid loss for RHA 0 wt% (No.2)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	22		
#2	1	30	73	2.10
#3	2	73		

Table A23 Fluid loss for RHA 0 wt% (No.3)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	18		
#2	1	30	78	2.20
#3	2	78		

Table A24 Fluid loss for RHA 5 wt% (No.1)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	27	57	2.05
#2	1	45		
#3	2	57		

Table A25 Fluid loss for RHA 5 wt% (No.2)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	28	57	2.05
#2	1	49		
#3	2	57		

Table A26 Fluid loss for RHA 5 wt% (No.3)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	29	58	2.05
#2	1	53		
#3	2	58		

Table A27 Fluid loss for RHA 10 wt% (No.1)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	41		
#2	1	63	78	2.40
#3	2	78		

Table A28 Fluid loss for RHA 10 wt% (No.2)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	40		
#2	1	64	79	2.41
#3	2	79		

Table A29 Fluid loss for RHA 10 wt% (No.3)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	40		
#2	1	63	77	2.40
#3	2	77		

Table A30 Fluid loss for RHA 15 wt% (No.1)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	47		
#2	1	69	91	2.12
#3	2	91		

Table A31 Fluid loss for RHA 15 wt% (No.2)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	47		
#2	1	70	93	2.13
#3	2	93		

Table A32 Fluid loss for RHA 15 wt% (No.3)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	47		
#2	1	69	92	2.13
#3	2	92		

Table A33 Fluid loss for RHA 20 wt% (No.1)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	20	110	13.0
#2	1	48		
#3	2	69		
#4	5	88		
#5	7 ½	97		
#6	10	100		

Table A34 Fluid loss for RHA 20 wt% (No.2)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	20	112	13.1
#2	1	49		
#3	2	70		
#4	5	88		
#5	7 ½	93		
#6	10	101		

Table A35 Fluid loss for RHA 20 wt% (No.3)

Reading	Time (min)	Fluid loss (cc)	Blow out (ml)	Time of blow out (min)
#1	½	20	112	13.1
#2	1	48		
#3	2	71		
#4	5	88		
#5	7 ½	95		
#6	10	100		

Filter cake data for all tested

Table A36 Filter Cake Description for RHA 0 wt% (No.1)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	31	55.65	firm
End	29		

Table A37 Filter Cake Description for RHA 0 wt% (No.2)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	31	55.55	firm
End	29		

Table A38 Filter Cake Description for RHA 0 wt% (No.3)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	31	55.60	firm
End	30		

Table A39 Filter Cake Description for RHA 5 wt% (No.1)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	30	54.85	firm
End	30		

Table A40 Filter Cake Description for RHA 5 wt% (No.2)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	29	54.79	firm
End	29		

Table A41 Filter Cake Description for RHA 5 wt% (No.3)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	29	54.82	firm
End	29		

Table A42 Filter Cake Description for RHA 10 wt% (No.1)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	31	61.80	firm
End	30		

Table A43 Filter Cake Description for RHA 10 wt% (No.2)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	31	61.80	firm
End	29		

Table A44 Filter Cake Description for RHA 10 wt% (No.3)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	29	61.81	firm
End	29		

Table A45 Filter Cake Description for RHA 15 wt% (No.1)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	31	60.50	firm
End	30		

Table A46 Filter Cake Description for RHA 15 wt% (No.2)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	30	60.50	firm
End	30		

Table A47 Filter Cake Description for RHA 15 wt% (No.3)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	30	60.51	firm
End	30		

Table A48 Filter Cake Description for RHA 20 wt% (No.1)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	31	63.40	firm
End	30		

Table A49 Filter Cake Description for RHA 20 wt% (No.2)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	30	63.40	firm
End	30		

Table A50 Filter Cake Description for RHA 20 wt% (No.3)

Temp (°C)		Thickness (mm)	consistency (hard/firm/mushy/gelled)
Start	30	63.41	firm
End	30		

Permeability data and parameter for all tested

Table A51 Permeability for RHA 0 wt%

Permeability								
Pc (psi)	P1 (psi)	P1 (atm)	Pb (atm)	Viscosity(n) (cp)	V (cm^3)	Time (sec)	Q (cm^3/sec)	K_gas (millidarcy)
560	40.05	2.72	1	0.0173	5	14.25	0.351	0.392
550	30.10	2.05	1	0.0173	5	17.03	0.294	0.509
540	20.09	1.37	1	0.0173	5	27.22	0.184	0.574
530	10.05	0.68	1	0.0173	2	24.31	0.082	0.644
3100	40.05	2.72	1	0.0173	5	15.31	0.327	0.365
3000	30.08	2.05	1	0.0173	5	22.22	0.225	0.390
3200	20.07	1.37	1	0.0173	5	37.03	0.135	0.422
3200	10.04	0.68	1	0.0173	2	30.53	0.066	0.514

Table A52 Permeability for RHA 5 wt%

Permeability								
Pc (psi)	P1 (psi)	P1 (atm)	Pb (atm)	Viscosity(n) (cp)	V (cm^3)	Time (sec)	Q (cm^3/sec)	K_gas (millidarcy)
620	40.04	2.72	1	0.0173	5	18.47	0.271	0.310
610	30.06	2.04	1	0.0173	5	24.71	0.202	0.360
600	20.09	1.37	1	0.0173	5	38.91	0.129	0.411
580	10.04	0.68	1	0.0173	2	33.15	0.060	0.485
3100	40.02	2.72	1	0.0173	5	26.13	0.191	0.219
3000	30.05	2.04	1	0.0173	5	34.66	0.144	0.257
3000	20.06	1.36	1	0.0173	2	21,28	0.094	0.302
2900	10.06	0.68	1	0.0173	2	46.19	0.043	0.347

Table A53 Permeability for RHA 10 wt%

Permeability								
Pc (psi)	P1 (psi)	P1 (atm)	Pb (atm)	Viscosity(n) (cp)	V (cm^3)	Time (sec)	Q (cm^3/sec)	K_gas (millidarcy)
510	40.09	2.73	1	0.0173	5	21.40	0.234	0.264
500	31.21	2.06	1	0.0173	2	12.16	0.164	0.288
610	20.37	1.39	1	0.0173	2	19.78	0.101	0.314
580	10.05	0.68	1	0.0173	2	41.41	0.048	0.384
3000	40.04	2.72	1	0.0173	5	30.54	0.164	0.185
2900	30.12	2.05	1	0.0173	5	43.34	0.115	0.203
3000	20.03	1.36	1	0.0173	2	28.66	0.070	0.222
2900	10.10	0.69	1	0.0173	2	61.50	0.033	0.257

Table A54 Permeability for RHA 15 wt%

Permeability								
Pc (psi)	P1 (psi)	P1 (atm)	Pb (atm)	Viscosity(n) (cp)	V (cm^3)	Time (sec)	Q (cm^3/sec)	K_gas (millidarcy)
570	40.08	2.73	1	0.0173	5	42.59	0.117	0.133
550	30.00	2.04	1	0.0173	5	57.28	0.087	0.155
530	20.04	1.36	1	0.0173	2	37.31	0.054	0.171
510	10.03	0.68	1	0.0173	2	73.41	0.027	0.218
3200	40.06	2.73	1	0.0173	5	50.97	0.098	0.111
3000	30.03	2.04	1	0.0173	5	71.16	0.070	0.124
2950	20.05	1.36	1	0.0173	2	46.00	0.043	0.139
3200	10.10	0.69	1	0.0173	2	101.10	0.020	0.157

Table A55 Permeability for RHA 20 wt%

Permeability								
Pc (psi)	P1 (psi)	P1 (atm)	Pb (atm)	Viscosity(n) (cp)	V (cm^3)	Time (sec)	Q (cm^3/sec)	K_gas (millidarcy)
650	40.07	2.73	1	0.0173	5	66.19	0.076	0.087
620	30.05	2.04	1	0.0173	5	63.01	0.079	0.143
610	20.04	1.36	1	0.0173	2	41.04	0.049	0.158
600	10.05	0.68	1	0.0173	2	88.58	0.023	0.183
3100	40.01	2.72	1	0.0173	5	56.07	0.089	0.103
3100	30.11	2.05	1	0.0173	5	78.28	0.064	0.115
3000	20.15	1.37	1	0.0173	2	50.60	0.040	0.127
3000	10.10	0.69	1	0.0173	2	111.21	0.018	0.145

Thickening time data for all tested

Table A56 Thickening Time for RHA 0 wt%

Reading	mixing time	time at testing	penetration (mm)						final setting time (min)
			0 min	60 min	120 min	180 min	240 min	300 min	
#1	11.42	11.45	40	40	38	32	29	3	330
#2			40	40	38	32	29	3	
#3			40	40	38	32	29	3	

Table A57 Thickening Time for RHA 5 wt%

Reading	mixing time	time at testing	penetration (mm)						final setting time (min)
			0 min	60 min	120 min	180 min	240 min	300 min	
#1	13.24	13.27	40	40	35	30	28	2	315
#2			40	40	35	30	28	2	
#3			40	40	35	30	28	2	

Table A58 Thickening Time for RHA 10 wt%

Reading	mixing time	time at testing	penetration (mm)						final setting time (min)
			0 min	60 min	120 min	180 min	240 min	300 min	
#1	14.21	14.27	40	40	35	30	1	-	270
#2			40	40	35	30	1	-	
#3			40	40	35	30	1	-	

Table A59 Thickening Time for RHA 15 wt%

Reading	mixing time	time at testing	penetration (mm)						final setting time (min)
			0 min	60 min	120 min	180 min	240 min	300 min	
#1	15.33	15.47	40	36	34	32	1	-	270
#2			40	36	34	32	1	-	
#3			40	36	34	32	1	-	

Table A60 Thickening Time for RHA 20 wt%

Reading	mixing time	time at testing	penetration (mm)						final setting time (min)
			0 min	60 min	120 min	180 min	240 min	300 min	
#1	16.19	16.27	40	40	40	35	34	1	315
#2			40	40	40	35	34	1	
#3			40	40	40	35	34	1	

Compressive Strength data for all tested

Table A61 Compressive Strength for RHA 0 wt% curing at 25°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	80.94	80.46	81.41	80.94	81.41
7	111.67	110.83	114.52	112.34	114.52
14	115.92	114.48	116.52	115.64	116.52
28	119.15	117.74	115.56	117.48	119.15
56	124.50	126.34	120.06	123.63	126.34

Table A62 Compressive Strength for RHA 0 wt% curing at 80°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	81.32	80.23	81.21	80.89	81.32
7	113.21	111.79	109.37	111.46	113.21
14	114.56	116.43	115.45	115.48	116.43
28	118.85	117.22	119.54	118.54	119.54
56	124.74	125.35	122.22	124.10	125.35

Table A63 Compressive Strength for RHA 5 wt% curing at 25°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	81.34	82.17	79.73	81.08	82.17
7	108.90	113.56	110.87	111.11	113.56
14	123.46	127.83	124.37	125.22	127.83
28	135.12	134.43	138.94	136.16	138.94
56	143.68	147.64	144.27	145.20	147.64

Table A64 Compressive Strength for RHA 5 wt% curing at 80°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	82.30	80.10	81.00	81.13	82.30
7	113.29	115.64	110.01	112.98	115.64
14	122.48	126.94	124.33	124.58	126.94
28	133.25	135.61	138.00	135.62	138.00
56	145.77	145.46	142.96	144.73	145.77

Table A65 Compressive Strength for RHA 10 wt% curing at 25°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	80.06	80.90	83.10	81.35	83.10
7	135.00	136.30	138.49	136.60	138.49
14	144.22	148.74	146.17	146.38	148.74
28	154.47	155.36	154.45	154.76	155.36
56	173.20	176.40	175.07	175.07	176.40

Table A66 Compressive Strength for RHA 10 wt% curing at 80°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	81.00	80.20	83.10	81.43	83.10
7	136.26	133.22	136.15	135.21	136.26
14	144.00	146.74	148.03	146.26	148.03
28	155.89	155.36	155.07	155.44	155.09
56	177.91	175.36	174.84	176.04	177.91

Table A67 Compressive Strength for RHA 15 wt% curing at 25°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	82.03	78.11	85.25	81.80	85.25
7	140.19	143.23	143.10	142.17	143.23
14	149.64	153.51	145.85	149.67	153.51
28	155.80	159.11	157.30	157.40	159.11
56	178.94	180.40	176.70	178.68	180.40

Table A68 Compressive Strength for RHA 15 wt% curing at 80°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	81.11	80.48	83.44	81.68	83.44
7	143.27	142.85	140.49	142.20	143.27
14	148.44	147.69	150.00	148.71	150.00
28	158.57	158.33	158.40	158.43	158.57
56	178.00	178.88	179.36	178.75	179.36

Table A69 Compressive Strength for RHA 20 wt% curing at 25°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	83.16	82.38	83.76	83.10	83.76
7	145.30	146.40	145.84	145.85	146.40
14	154.28	151.94	150.50	152.24	154.28
28	160.81	157.10	158.43	158.78	160.81
56	179.51	183.40	185.10	182.67	185.10

Table A70 Compressive Strength for RHA 20 wt% curing at 80°C

DAYS	STRENGTH (KN)			AVERAGE	HIGH
	#1	#2	#3		
3	84.00	80.67	83.41	82.69	84.00
7	144.73	144.68	145.07	144.84	145.07
14	151.66	150.95	151.74	151.45	151.74
28	159.98	160.01	158.85	159.61	160.01
56	181.81	180.93	184.15	182.30	184.15

BIOGRAPHY

Mr. Nattapon Kaeso was born on the 20th of December 1988 in Samutsongkram, Thailand. He earned his high school diploma in science-math from Prommanusorn School in 2006 and he earned his Bachelor's Degree in Geotechnology Engineering from Suranaree University of Technology in 2010. After graduation, he continued with his master's degree in the School of Geotechnology, Institute of Engineering at Suranaree University of Technology (SUT) in Petroleum Engineering Program. During 2011-2015 (SUT), he was a teaching assistant and laboratory assistant at SUT. His strong background is in the areas of well completion and well production.